NASA Technical Memorandum 108523
Volume IV

The Microgravity Research Experiments (MICREX) Data Base

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November 1996

Acknowledgments

The NASA Marshall Space Flight Center Microgravity Research Experiments (MICREX) data base has been under the sponsorship of two NASA Headquarters' divisions: the Microgravity Science and Applications Division (MSAD) and the Office of Commercial Programs (OCP). The creation and upkeep of the data base (and completion of this document) would not have been possible if it had not been for the vision and efforts of the following personnel: Gary Martin/NASA Headquarters, Charles Schafer/MSFC, EP13, Charles Baugher/MSFC, ES71, Tim Baldridge/MSFC, ES94, Fred Leslie/MSFC, ES71, Ken Taylor (formerly MSFC/PS05), Tommy Bannister (formerly the Center for Space and Advanced Technology (CSAT)), Randall Stanley (formerly CSAT), Kervin Evans (formerly MSFC/ES42), Anthony Doucette (formerly Wyle Laboratories), George Fichtl (formerly MSFC/JA81), Robert Snyder/MSFC, ES71, and Bob Naumann (formerly MSFC/ES71). Technical editing of this document was performed by Tauna Moorehead (formerly MSFC/ES01) and Judy Maples (DPTI). Michael McMurray (Management Services Incorporated) assisted with the preparation of the figures.

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CHAPTER 17

SYSTEMS EXHIBITING A MISCIBILITY GAP

Principal Investigator(s): Reger, J. L. (1)
Co-Investigator(s): Hammel, R. L. (Program Manager/TRW) (2), Wuenscher, H. (3), Yates, I. C. (Project Engineer) (4)
Affiliation(s): (1) During Apollo 14: TRW Systems Group, Redondo Beach, California, Currently: Unknown; (2) TRW Systems Group, Redondo Beach, California; (3,4) During Apollo 14: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), Huntsville, Alabama; (3) Currently: Unknown; (4) Currently: Retired

Experiment Origin: USA
Mission: Apollo 14
Launch Date/Expt. Date: February 1971
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Science Demonstration, Apollo Command Module Payload
Processing Facility: A hand-held electric heater which accepted sealed capsules containing sample materials
Builder of Processing Facility: Heater and solidification capsules were provided to investigators by NASA Marshall Space Flight Center, Huntsville, Alabama.

Experiment:
Composite Casting- Part III: Paraffin-Based Immiscible Samples

<Note: A total of eleven samples were processed during the Composite Casting Experiment. The evaluation of three of the samples (denoted by the investigators as "Part III" of the experiment) is discussed here. Discussion of the other eight samples can be found in Chapter 5 under Fabiniak, Apollo 14; Peters, Apollo 14; Steurer, Apollo 14.>

This Apollo 14 composite casting experiment (Part III) was the first in a series of investigations designed by Reger et al. to study the solidification of immiscible alloys under low-gravity conditions. The specific objective of the experiment was to examine the potential for forming unique immiscible materials dispersions in the reduced gravity environment.

Three experiment capsules were prepared prior to the Apollo 14 flight. The first contained immiscible liquids, the second contained immiscible liquids and a gas, and the third contained immiscible liquids and a solid. During the mission, each sample was placed in an electric heater and warmed for 10 minutes. The heater was shaken by hand to mix the materials. Solidification took place when the heater and sample were placed in a heat sink.

TRW conducted post-flight evaluation of the three samples and compared them to Earth-processed control samples.

The first sample was composed of paraffin and sodium acetate. Evaluation of the ground-based sample indicated clear segregation of the two liquids. In contrast, the flight sample exhibited only a partial segregation; some dispersion of sodium acetate in paraffin and paraffin in sodium acetate could be observed.

The second sample was composed of paraffin, sodium acetate, and argon. The ground-based sample experienced complete segregation of paraffin and sodium acetate. In contrast, the flight sample experienced an almost complete dispersion of paraffin and sodium acetate. It was noted that an appreciable gas dispersion was not observed in the flight sample.

The third sample was composed of paraffin, sodium acetate, and tungsten microspheres. The ground-based sample experienced a high degree of segregation. In contrast, the flight sample experienced three types of sodium acetate, paraffin, and tungsten dispersions.

Reportedly, none of the three samples exhibited homogeneous material or phase distributions.

Additional information concerning the detailed analyses of each sample can be found in the references listed below.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Immiscible Fluids, Melt and Solidification, Segregation, Density Difference, Liquid Mixing, Dispersion, Liquid/Liquid Dispersion, Liquid/Gas Dispersion, Solid/Liquid/Gas Dispersion, Particle Dispersion, Multiphase Dispersion, Multiphase Media, Liquid/Gas Interface, Solid/Liquid Interface, Liquid/Liquid Interface, Stirring of Components

Number of Samples: three
Sample Materials: (1) 50 volume % paraffin and 50 volume % sodium acetate (a tungsten mixing pellet was included); (2) 40 volume % paraffin, 40 volume % sodium acetate trihydrate, 20 volume % argon (a tungsten mixing pellet was included); (3) 40 volume % paraffin, 40 volume % sodium acetate trihydrate, and 20 volume % 100-micron diameter tungsten microspheres (a tungsten mixing pellet was included).
(Ar*,W*)

Container Materials: unknown

Experiment/Material Applications:
In addition to examining basic solidification phenomenon in low gravity, this experiment sought to determine if immiscible mixtures of liquids, liquids and solids, or liquids and gas would disperse uniformly in low gravity.

References/Applicable Publications:
(1) Yates, I. C.: Apollo 14 Composite Casting Demonstration. In Process Engineering Research at MSFC, Research Achievements Review, Vol. IV, Report No. 7, Marshall Space Flight Center, Alabama, NASA TM X-64723, February 1973. (post-flight)

(2) Reger, J. L.: Low Gravity Processing of Immiscible Materials. 23rd International Astronautical Federation, International Astronautical Congress, Vienna, Austria, October 8-15, 1972, 9 pp. (post-flight)

(3) Yates, I. C., Jr.: Apollo 14 Composite Casting Demonstration-Final Report. NASA TM X-64641, October 1971.

(4) Reger, J. L. and Yates, I. C.: Preparation of Metallurgical Properties of Low Gravity Processed Immiscible Materials. Presented at the AIAA 12th Aerospace Sciences Meeting, January 30-February 1, 1974, Washington, D.C.

(5) Input received from Co-Investigator R. L. Hammel, May 1991.

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Co-Investigator(s): Larson, D. J., Jr. (officially not a Co-Investigator) (2)
Affiliation(s): (1) During Skylab: TRW Systems Group, Redondo Beach, California, Currently: Unknown; (2) Grumman Aerospace Corporation, Bethpage, New York

Experiment Origin: USA
Mission: Skylab, SL-3, Second Manned Mission
Launch Date/Expt Date: September 1973 (month experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA) area, Skylab Manned Environment
Processing Facility: Multipurpose Electric Furnace System (MEFS)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Immiscible Alloy Compositions (M557)

When certain multi-component metallic systems are cooled below a particular temperature (the consulate temperature), they separate because of compositional density differences of system components (similar to the behavior of oil and water). When such immiscible systems are solidified on Earth, useful materials exhibiting a matrix containing a fine, homogeneous dispersion of the second phase rarely result.

This Skylab SL-3 experiment was the second in a series of investigations designed by Reger et al. to study the solidification of immiscible alloys under low-gravity conditions (see Reger, Apollo 14). The specific objectives of the experiment were to determine (1) if gravity-induced sedimentation and buoyancy would be reduced during space solidification of the alloys and (2) if the resulting material would exhibit a fine dispersion of the minority phase.

Prior to the mission, three stainless steel sample cartridges were prepared. Each cartridge was configured with three ampoules which each contained one sample material (nine total samples): (1) Pb-45.06 wt.% Zn-9.89 wt.% Sb, (2) Au-23.15 wt.% Ge, and (3) Pb-14.80 wt.% In-15.00 wt.% Sn. Samples of type (1) and (2) were contained in stainless steel ampoules and samples of type (3) were contained in quartz ampoules.

During the SL-3 mission, the samples in the three cartridges were processed simutaneously using the M-518 Multipurpose Electric Furnace. Samples of type (1) and (2) (Pb-Zn-Sb and Au-Ge) were processed isothermally: heated to 720 ^{o}C and soaked at this temperature for 4 hours before passive cooling took place. (This soak temperature was selected because it was above the consulate temperature of the Pb-Zn-Sb sample.) The soak period was sufficiently long to allow diffusion and complete mixing of the elements. Samples of type 3 (Pb-In-Sn) were directionally solidified by heating the material such that one end was not allowed to melt. Thus, when the furnace was cooled, the sample solidified directionally from the cold end. Two samples of each alloy (one solidified vertically and the other horizontally) were processed on Earth as control samples and used for comparison.

Post-flight analysis revealed that reduced-gravity processing of the Au-Ge samples resulted in a more random distribution of Ge than in the corresponding 1-g specimens. When the space processed specimens were compared to a sample cooled at a much faster rate during KC-135 low-gravity aircraft experiments, it was concluded that solidification rate has a significant effect on the resulting microstructure. X-ray diffraction studies of the low-gravity specimens revealed diffraction lines of unknown origin: these lines did not match with known diffraction lines from the system. Studies indicated that the Skylab specimens superconducted at 1.5 K (weak signals) but that the 1-g specimens exhibited no superconducting behavior. Resistivity measurements (Leeds four point probe) indicated a more uniform resistivity for the low-gravity samples than that of the 1-g samples.

The 1-g processed Pb-Zn-Sb samples showed significant segregation, whereas the low-gravity samples showed a more uniform dispersion. Further examination of the Skylab samples revealed that the Zn was the primary matrix material with Pb as the dispersant. Sb was primarily associated with the Zn. This was not the case in the 1-g sample. As with the Au-Ge specimens, X-ray diffraction studies showed lines which could not be matched with those caused by the elements or related compounds. Superconductivity measurements indicated that both 1-g and low-gravity specimens exhibited a transition temperature of 7.2 K but the space-processed samples also showed a second transition at 9.2 K. The resistivity of the low-gravity samples was reportedly more uniform than that of the 1-g sample.

In general, the microstructure of the directionally solidified portion of the Pb-Sn-In, Skylab samples were "...finer and of better quality..." (3, p. 136) than those processed in 1-g. X-ray diffraction studies indicated (unlike the isothermal samples) that there was no appreciable difference between the 1-g and low-gravity processing. There was a slight increase in the magnetic

coercive strength of the low-gravity samples over the 1-g processed specimens. Further examination indicated that the space-processed, Pb-Sn-In samples "...were found to have thinner lamellae (...[about]... 1 micron) than samples processed in a ground-based laboratory. Increased magnetic flux pinning by the Sn-rich phase was indicated from the small sections of the Skylab samples ([about] 5%) which experienced the most beneficial solidification conditions. Thinner lamellae, and hence, increased flux pinning, could be obtained by increasing the solidification rate." (9, p. 6)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Ternary Systems, Melt and Solidification, Isothermal Processing, Directional Solidification, Density Difference, Separation of Components, Sedimentation, Segregation, Precipitation, Diffusion, Diffusive Mass Transfer, Liquid Mixing, Buoyancy Effects, Dispersion, Homogeneous Dispersion, Liquid/Liquid Dispersion, Multiphase Media, Solid/Liquid Interface, Liquid/Liquid Interface, Thermal Soak, Thermal Gradient, Passive Cooling, Solidification Rate, Sample Microstructure, Lamellar Structure, Superconductivity, Magnetic Properties

Number of Samples: nine
Sample Materials: Two isothermal samples: (1) Au-23.15 wt.% Ge and (2) Pb-45.06 wt.% Zn-9.89 wt.% Sb; one directional sample: Pb-14.80 wt.% Sn-15.00 wt.% In.
(Au*Ge*, Pb*Zn*Sb*, Pb*Sn*In*)
Container Materials: isothermal samples: stainless steel; gradient samples: quartz

Experiment/Material Applications:
Immiscible systems are a unique class of material which cannot be successfully processed in large quantities on Earth because of the separation of the liquid constituents before solidification. Successful processing in space, without compositional sedimentation due to gravity, could result in materials with unique electronic or optical properties.

Au-Ge and Pb-Zn-Sb were selected for the isothermal experiments because (1) Au-Ge alloys exhibit almost complete solid state immiscibility and (2) Pb-Zn-Sb exhibits a miscibility gap below a consulate temperature which could be exceeded by the experimental

apparatus.

Above the consulate temperature the Pb-Sn-Sb liquid alloy exists as a single phase. The Pb-Sn-In sample was selected as the directionally solidified sample because Pb precipitates out as a second phase and can possibly be preferentially oriented.

References/Applicable Publications:

(1) Naumann, R. J. and Herring H. W.: Experiment M557, Immiscible Alloy Composites. In Materials Processing in Space: Early Experiments, NASA SP-443, pp. 68-69. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements; post-flight)

(3) Reger, J. L.: Experiment No. M-557 Immiscible Alloy Composites. In Proceedings the Third Space Processing Symposium, Skylab Results, Vol. I, April 30-May 1, 1974, NASA Marshall Space Flight Center, Alabama, pp. 133-158. (post-flight)

(4) Experiment M558-Radioactive Tracer Diffusion. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-56 - 5-62. (post-flight)

(5) M518-Multipurpose Electric Furnace System. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-42 - 5-56. (processing facility)

(6) Multipurpose Electric Furnace (M518). In MSFC Skylab Mission Report-Saturn Workshop. NASA TM X-64814, October 1974, pp. 12-46 - 12-49. (processing facility)

(7) Immiscible Alloy Compositions (M557). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-49 - 12-50. (post-flight)

(8) Naumman, R. J. and Mason, D.: Immiscible Alloy Compositions. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1989, pp. 22-23. (post-flight)

(9) Anderson, W. T. and Reger, J. L.: Superconducting Properties of Pb-Sn-In Alloys Directionally Solidified Aboard Skylab. In AIAA 10th Thermophysics Conference, Denver, Colorado, May 27-29, 1975, AIAA Paper No. 75-694. (post-flight)

(10) Reger, J. L. and Yates, I. C.: Preparation and Metallurgical Properties of Low Gravity Processed Immiscible Materials. In AIAA 12th Aerospace Sciences Meeting, Washington, D.C., January 30-February 1, 1974, AIAA Paper No. 74-207. (post-flight)

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Experiment Origin: USA
Mission: Skylab, SL-4, Third Skylab Manned Mission
Launch Date/Expt Date: December 1973 (month experiments were completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA) area, Skylab Manned Environment
Processing Facility: Multipurpose Electric Furnace System (MEFS)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Immiscible Alloy Compositions (M557)

This Skylab SL-4 experiment was the third in a series of investigations designed by Reger et al. to study the solidification of immiscible alloys under low-gravity conditions (see Reger, Apollo 14; Skylab SL-3). The specific objective of the investigation was to determine the effects of the space environment on immiscible systems which separate during processing on Earth.

During the SL-4 mission, two immiscible alloys (Au-Ge and Pb-Zn-Sb) were solidified isothermally and one immiscible alloy (Pb-Sn-In) was solidified directionally within the M-518 Multipurpose Electric Furnace.

The sample compositions and processing parameters appear to have been identical to those employed during the Skylab SL-3 experiment (see Reger, SL-3).

<Note: Reference (4) states that the Skylab SL-4 experiment "...was successfully performed from DOY [Day of Year] 355, 1900 GMT [Greenwich Mean Time] through DOY 357, 2040 GMT." (4, p. 5-60) However, no publications that discuss the exact experiment procedure or analysis of these samples could be located at this time.>

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Ternary Systems, Melt and Solidification, Isothermal Processing, Directional Solidification, Density Difference, Separation of Components, Sedimentation, Segregation, Buoyancy Effects, Diffusion, Dispersion, Homogeneous Dispersion, Liquid/Liquid Dispersion, Thermal Gradient

Number of Samples: nine
Sample Materials: Two isothermal samples: (1) Au-23.15 wt.% Ge and (2) Pb-45.06 wt.% Zn-9.89 wt.% Sb; one directional sample: Pb-14.80 wt.% Sn-15.00 wt.% In.
(Au*Ge*, Pb*Zn*Sb*, Pb*Sn*In*)
Container Materials: isothermal samples: stainless steel; gradient samples: quartz
(Si*O*)

Experiment/Material Applications:
See Reger, Skylab SL-3.

References/Applicable Publications:

(1) Naumann, R. J. and Herring H. W.: Experiment M557, Immiscible Alloy Composites. In Materials Processing in Space: Early Experiments, NASA SP-443, pp. 68-69. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements; post-flight)

(3) Reger, J. L.: Experiment No. M-557 Immiscible Alloy Composites. In Proceedings the Third Space Processing Symposium Skylab Results, Vol. I, April 30-May 1, 1974, NASA Marshall Space Flight Center, Alabama, pp. 133-158. (post-flight)

(4) Experiment M557-Immiscible Alloy Compositions. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-57 - 5-62. (post-flight)

(5) M518-Multipurpose Electric Furnace System. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-42 - 5-56. (processing facility)

(6) Multipurpose Electric Furnace (M518). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-46 - 12-49. (processing facility)

(7) Immiscible Alloy Compositions (M557). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-49 - 12-50. (post-flight)

(8) Reger, J. L. and Yates, I. C.: Preparation and Metallurgical Properties of Low Gravity Processed Immiscible Materials. In AIAA 12th Aerospace Sciences Meeting, Washington, D.C., January 30-February 1, 1974, AIAA Paper No. 74-207. (post-flight)

(9) Anderson, W. T. and Reger, J. L.: Superconducting Properties of Pb-Sn-In Alloys Directionally Solidified Aboard Skylab. In AIAA 10th Thermophysics Conference, Denver, Colorado, May 27-29, 1975, AIAA Paper No. 75-694. (post-flight)

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Experiment Origin: USA
Mission: Skylab, SL-4, Third Skylab Manned Mission
Launch Date/Expt. Date: January 1974 (month experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Science Demonstration, Skylab Manned Environment
Processing Facility: Hand held apparatus: polycarbonate clear vials filled with water and oil (brass nut included to mix liquid)
Builder of Processing Facility: NASA Marshall Space Flight Center, Space Sciences Laboratory, Huntsville, Alabama

Experiment:
Immiscible Liquids (SD19 TV102)

This Skylab SL-4 experiment was the first in a series of investigations designed by Lacy et al. to study the low-gravity behavior of immiscible systems. The primary objectives of the experiment were to (1) visibly examine the stability of two immiscible liquids, and (2) investigate the rate of coalescence of the liquids after dispersion.

The experimental apparatus used during the Skylab experiment consisted of three transparent plastic (polycarbonate) vials each containing different mixtures of degassed Krytox 143 AZ oil and degassed red-colored water. The first vial contained 25 vol.% oil, the second 50 vol.% oil, and the third 75 vol.% oil. (The employed fluids had different densities and their fluid properties had been well characterized.) All three vials were mounted in a single stainless steel frame.

The fluids within the vials assembly could be separated when an astronaut took hold of a string attached to the frame and swung the assembly in a circular orbit. The fluids could then be dispersed when the frame was vigorously shaken by the astronaut. (A small brass nut included in each vial aided the dispersion process.) A card with parallel black lines was placed behind the transparent vials to aid in analysis of the liquid separation.

The Skylab experimental procedure consisted of (1) centrifuging the vials to separate the liquids, (2) shaking the vials to obtain a dispersion, (3) video-taping the emulsions over a period of several minutes (immediately after mixing) and (4) sequentially photographing (35 mm) the liquids over a 10-hour period (to record the coalescence process).

During similar ground-based experiments, high-speed motion pictures were used to photograph the separation process.

Post-flight, the relative stability of the low-gravity and 1-g dispersions were obtained using two methods: (1) determining the volume fraction of separation between the two liquids (using the parallel lines of the card placed behind the vials) and (2) measuring the red color density of the 35 mm transparencies as a function of time (photodensitometry).

Results from the volume fraction determination indicated the emulsions of the 1-g specimens were highly unstable: significant separation occurred in the 25% oil mixture after 0.1 second with a comparable separation in the 75% oil mixture after 0.8 second. Complete separation occurred in both of the 1-g samples after 2 and 10 seconds, respectively. (The 1-g results from the 50% oil mixture were intermediate between the 25% and 75% oil mixtures.) All low-gravity specimens were less separated after 10 hours than the 1-g, 25% oil mixture after 0.1 second. It was concluded that the coalescence rate for the Skylab specimens was reduced to 3×10^{-6} times that observed on Earth.

The photodensitometry studies also indicated that the low-gravity dispersions were stable during the 10-hour test period in an isothermal environment.

Comparison of the Krytox oil-water system to immiscible liquid metal systems was made using Stokes law and known fluid parameters. It was determined that the major differences between these material systems was "...the much larger surface tension found for liquid metals. An increase in surface tension will require an increase in the kinetic energy of colliding and subsequently coalescing particles to overcome the larger surface energies involved. Thus, it is expected that emulsions of metallic liquids should be as stable as the Krytox-water system." (1, p. 6)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Fluids, Binary Systems, Isothermal Processing, Liquid Mixing, Emulsion, Density Difference, Dispersion, Drops, Droplet Dispersion, Liquid/Liquid Dispersion, Stability of Dispersions, Drop Coalescence, Surface Tension, Surface Energy, Separation of Components, Bubble Removal, Liquid/Liquid Interface, Centrifuge, Buoyancy Effects Diminished

Number of Samples: three
Sample Materials: three different dispersions of oil and red-colored water (25, 50, and 75 %volume degassed Krytox 143 AZ oil)
Container Materials: polycarbonate plastic vials

Experiment/Material Applications:
Oil and water were chosen as the experimental liquids because they represent a classic immiscible fluid system. The fluids are also transparent and well characterized.

References/Applicable Publications:

(1) Lacy, L. L. and Otto, G. H.: The Stability of Liquid Dispersions in Low Gravity, AIAA Paper 74-1242. In American Institute of Aeronautics and Astronautics and American Geophysical Union, Conference on Scientific Experiments of Skylab, Huntsville, Alabama, October 30-November 1, 1974, 8 pp., NASA supported research. (post-flight)

(2) Lacy, L. L. and Otto, G. H.: Stability of Liquid Dispersions in Low Gravity. In Material Sciences in Space with Applications to Space Processing, AIAA Progress Series in Astronautics and Aeronautics, edited by Leo Steg, Vol. 52, p. 495, 1977.

(3) Lacy, L. L. and Otto, G. H.: The Electrical Properties of Zero-Gravity Processed Immiscibles. AIAA Paper 74-208, AIAA 12th Aerospace Sciences Meeting, Washington, D.C., January 30-February 1, 1974.

(4) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements)

(5) "TV102- Immiscible Liquids." In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 7-11 to 7-13. (post-flight)

(6) Naumann, R. J. and Herring, H. W.: "Experiment TV102, Immiscible Liquids." In Materials Processing in Space, Early Experiments, NASA SP-443, 1980. (post-flight)

(7) Bannister, T. C.: Skylab III and IV Science Demonstrations Preliminary Report. NASA TM X-64835, pp. 8-13. (post-flight)

(8) Immiscible Liquids (SD19-TV102). In MSFC Skylab Mission Report - Saturn Workshop, NASA TM X-64814, October 1974, p. 12-89.

(9) Input received from Principal Investigator, L. L. Lacy, July 1993.

(10) Input received from Co-Investigator G. H. Otto, August 1993.

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Experiment Origin: USA
Mission: ASTP
Launch Date/Expt. Date: July 1975
Launched From: NASA Kennedy Space Center, Florida
Payload Type: ASTP Docking Module Payload
Processing Facility: Multipurpose electric gradient furnace located in the ASTP docking module
Builder of Processing Facility: Unknown, possibly Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Monotectic and Synthetic Alloys (MA-044)

This ASTP experiment was the second in a series of investigations designed by Lacy et al. to study the low-gravity behavior of immiscible systems (see Lacy, Skylab SL-4). The specific objectives of the space research were to (1) study the phase segregation of the immiscible binary system Pb-Zn and (2) determine the effects of low-gravity processing on the microstructural homogeneity and stoichiometry of an AlSb semiconductor compound.

Prior to the mission, three identical samples of the following two compositions were prepared for the experiments: (1) 20 at.% Pb - 80 at.% Zn and (2) 50 at.% Al - 50 at.% Sb (intermetallic composition). Three stainless steel cartridges, each containing two graphite ampoules (one sample per ampoule), held the six samples. Each AlSb sample was contained in an ampoule 11.9 mm in diameter, 29.0 mm long and each Pb-Zn sample was contained in an ampoule 9.9 mm in diameter, 19.0 mm long. The three cartridges were configured into three cavities within the ASTP Multipurpose Electric Furnace. Each of these three cavities had a short hot zone and longer gradient zone.

During the first 200 minutes of the space experiments (1) the AlSb samples were heated to 1403 K (50 K above the melt temperature) and (2) the Pb-Zn samples were heated to 1108 K (40 K above the consulate temperature). After the desired temperatures were reached, the samples were subjected to an isothermal soak for 1 hour. This soak was followed by a 6.5 hour cool-down period of all samples. (See Reference (4) for a plot of the samples' thermal history.) Ground-based tests were also per-

formed using similar samples and thermal histories.

Post-flight characterization of the samples included (1) metallography, (2) quantitative microstructural analysis, (3) scanning electron microscopy, (4) energy dispersive X-ray analysis, (5) electrical resistivity measurements, (6) X-ray diffraction, (7) chemical analysis, and (8) ion-microprobe mass analysis.

Analysis of the AlSb samples revealed that the low-g samples had significantly more macroscopic and microscopic homogeneity than the 1-g samples. Specifically, the low-g samples contained 4 to 20 times less unwanted secondary phase than the 1-g material. Major grains of an Al-rich or Sb-rich phase were present in the 1-g samples while the space-processed material contained only small amounts of an Al-rich phase. Analysis of the diffusion and liquid-state homogenization in the 1-g and low-gravity samples indicated that convection induced by gravity resulted in compositional and microstructural inhomogeneity during solidification.

Although the Pb-Zn material was soaked 40 K above the reported consulate temperature, incomplete liquid-state mixing was observed in the low-gravity samples. A fine particle dispersion of Pb in the Zn matrix was indicated, but the majority of the Pb remained in its original position. It was suspected that the accepted phase diagram for the Pb-Zn system may have contained a significant inaccuracy. Further research (see Reference (10)) indicated that the accepted miscibility curve between 20 at.% Zn and 70 at.% Zn is as much as 20 ^{o}C too low. Also, the quasi-solid-state diffusion coefficient of Pb into Zn was found to be 2.4 x 10^{-6} cm^2/sec, which was about two orders of magnitude smaller than that expected from 1-g experiments. Therefore, it is possible that the melt was never completely mixed during the low-gravity experiment.

<Note: Further details of the analysis and results for all samples may be located in Reference (4).>

Key Words: Systems Exhibiting a Miscibility Gap, Melt and Solidification, Directional Solidification, Immiscible Alloys, Monotectic Compositions, Intermetallics, Binary Systems, Diffusion, Diffusion Coefficient, Segregation, Phase Segregation, Buoyancy-Driven Convection, Composition Distribution, Dispersion, Liquid/liquid Dispersion, Particle Dispersion, Liquid Mixing, Sample Homogeneity, Multiphase Media, Liquid/Liquid Interface, Solid/Liquid Interface, Thermal Soak, Thermal Gradient, Sample Microstructure, Grain Structure, Superconductivity, Semiconductors, Electronic Materials, Incomplete Sample Processing

Number of Samples: six
Sample Materials: three samples each of (1) 20 at.% Pb - 80 at.% Zn and (2) 50 at.% Al - 50 at.% Sb (AlSb intermetallic)
(Pb*Zn*, Al*Sb*)
Container Materials: graphite ampoules with stainless steel cartridges
(C*)

Experiment/Material Applications:
The Pb-Zn material (like the AlSb alloy) was chosen because significant specific gravity differences between the components prevent adequate mixing on Earth. Reducing the effects of gravity could lead to a fine dispersion of superconducting Pb particles within the Zn matrix. (Pb-Zn is also a good material for modeling the behavior of immiscible systems.)

AlSb may be a more efficient solar cell material than commonly-used silicon.

References/Applicable Publications:

(1) Ang, C. Y. and Lacy, L. L.: Gravitational Influences on the Liquid-State Homogenization and Solidification of Aluminum Antimonide. Metallurgical Transactions A, Vol. 10A, May 1979.

(2) Lacy, L. L. and Ang, C. Y.: Low Gravity Homogenization and Solidification of Aluminum Antimonide. In Material Sciences in Space with Applications to Space Processing, AIAA Progress Series in Astronautics and Aeronautics, Edited by Leo Steg, Vol. 52, p. 523, 1977.

(3) Lacy, L. L. and Trahan, J. F.: Determination of Liquid Phase Immiscibility in the Lead Zinc System. Material Science and Engineering, Vol. 33, p. 249, 1978.

(4) Lacy, L. L. and Ang, C. Y.: Monotectic and Synthetic Alloys. In Apollo Soyuz Test Project Summary Science Report, Vol. 1, NASA SP-412, pp. 403-428, 1977. (post-flight)

(5) Boese, A., McHugh, J., and Seidensticker, R.: Multipurpose Electric Furnace. In Apollo-Soyuz Test Project Summary Science Report, Vol. I, NASA SP-412, pp. 353-365 (post-flight)

(6) Lacy, L. L. and Ang, C. Y.: Monotectic and Synthetic Alloys Experiment MA-044. In Apollo-Soyuz Test Project-Composite of MSFC Final Science Report, NASA TM X-73360, January 1977, pp. IV-1 - IV-51. (post-flight)

(7) Naumann, R. J. and Mason, E. D.: Monotectic and Synthetic Alloys. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, pp. 58-59. (post-flight)

(8) Input received from Principal Investigator L. L. Lacy, July 1993.

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Co-Investigator(s): Unknown
Affiliation(s): (1) Grumman Aerospace Corporation, Bethpage, New York

Experiment Origin: USA
Mission: ASTP
Launch Date/Expt. Date: July 1975
Launched From: NASA Kennedy Space Center, Florida
Payload Type: ASTP Docking Module Payload
Processing Facility: Multipurpose Electric Furnace (gradient furnace) located in the ASTP docking module
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Zero-G Processing of Magnets (MA-070)

Gravity-induced convection currents, produced during the directional solidification of eutectic alloys on Earth, contribute to microstructural imperfections in the rod or lamellar structure. In the low-gravity environment (1) the strength of such currents should be reduced and (2) the processing of eutectics with increased magnetic coercivity may be possible.

This experiment was the first in a series of investigations designed by Larson to study the directional solidification of magnetic components in the space environment.

The experiment was performed in three furnaces, each of which housed a single cartridge. Within each cartridge was a "Type 1 Ampoule," a "Type 2 Ampoule," and a "Type 3 Ampoule." Reportedly, (1) each Type 1 Ampoule was constructed of pyrolytic boron nitride and contained 50 at.% Bi, 50 at.% Mn, (2) each Type 2 Ampoule was constructed of pyrolytic boron nitride and contained 8 at.% Ce, 92 at.% Cu and Co, and (3) each Type 3 Ampoule was constructed of fused silica and contained 97.8 at.% Bi, 2.2 at.% Mn. (The Type 3 Ampoules were backfilled with argon to "...suppress the possibility of thermal cavitation of the bismuth-rich liquid.") (1, p. 456)

The experiment operating scenario was planned based on thermal results from a prototype test. "It was anticipated that the thermal environment for ampoule 1... would be essentially isothermal over the temperature range of solidification. Ampoule 2 was expected to be exposed to a thermal gradient of 30... [plus or minus] 2 K/cm and ampoule 3 to a thermal gradient of 60 [plus or minus] 3 K/cm." (1, p. 451) Reportedly, the samples were heated to the operating temperature, held at this temperature for

45 minutes, and then solidified by helium injection.

Reportedly, the flight samples were "...exposed to conditions substantially different from planned conditions...." (1, p. 451). For example (1) "The temperature gradient for ampoule 2... [varied] from approximately 30 K/cm at the onset of directional solidification to 23.5 K/cm at the conclusion of solidification" (1, p. 451) and (2) "The thermal gradient [in ampoule 3] during the period of maximum superheating was 80 K/cm; however, during the period of solidification, it varied from only 10.9 K/cm at the onset to 8.9 K/cm at the conclusion." (1, p. 451).

A detailed discussion of the objectives and results of each ampoule type in terms wetting behavior was presented in Reference (1). In general, it was reported that "Fluid static configurations in a low-g environment were appreciably different than in one-g, but were found to agree well with theory." (1, p. 469).

Post-flight it was reported that "...directional solidification was not achieved in the type 2 samples [Type 2 Ampoules], and the magnetic properties of the ground-based and flight samples were essentially the same." (1, p. 463) However, "The samples of 50 atomic percent (at.%) Bi-50 at.% Mn [Type 1 Ampoules] solidified in the low g environment demonstrated a substantial improvement in the macroscopic chemical homogeneity. The Bi/MnBi directionally solidified eutectic flight samples exhibited markedly superior magnetic properties. Intrinsic coercive strengths in excess of 14 722 kA/m (185 kOe) have been measured in the low g processed samples at a temperature of 77K. This strength exceeds the maximum previously published value (8992 kA/m (113kOe)) by 64 percent. The average value of inductance was improved by 76 percent and the energy product by 57 percent." (1, p. 449) Further discussions of the magnetic analysis are presented in Reference (1).

Among the experimental conclusions reported were:
(1) "Macroscopic chemical segregation due to gravitationally dependent buoyancy forces is minimal in low gravity." (1, p. 469)

(2) "The number and size distribution of orbitally processed primary crystals is significantly different in the flight samples." (1, p. 469)

(3) "The intrinsic coercive strengths of as-grown low-g MnBi/Bi eutectic samples greatly exceed any values previously reported for this magnetic composite (>60 percent)." (1, p. 469)

(4) "The solidification product from the orbital processing of the Bi/MnBi faceted rod eutectics differs in particle size and shape, lattice parameter, and magnetic properties from equivalently processed terrestrial samples." (1, p. 469)

Experiments related to this research can be found under Pirich, SPAR 6 and 9 (this chapter).

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Eutectics, Faceted Eutectics, Magnetic Composites, Binary Systems, Ternary Systems, Melt and Solidification, Directional Solidification, Thermal Gradient, Isothermal Processing, Thermal Soak, Superheating, Quench Process, Segregation, Buoyancy Effects, Particle Dispersion, Particle Size Distribution, Wetting, Wetting of Container, Sample Homogeneity, Thermosolutal Convection, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Solid/Liquid Interface, Sample Microstructure, Lamellar Structure, Rod Structure, Magnetic Properties, Brittleness, Processing Difficulties

Number of Samples: nine
Sample Materials: Type 1 Ampoules: 50 at.% Bi, 50 at.% Mn; Type 2 Ampoules: 8 at.% Ce, 92 at.% Cu and Co; Type 3 Ampoules: 97.8 at.% Bi, 2.2 at.% Mn.
(Mn*Bi*, Ce*Cu*Co*)
Container Materials: Ampoule Types 1 & 2: pyrolytic boron nitride; Ampoule Type 3: fused silica (quartz)
(B*N*, Si*O*)

Experiment/Material Applications:
"Because of the brittleness and reactivity of most magnetic compounds, powder metallurgy techniques principally have been used to fabricate high-coercive strength magnets. Casting and solidification techniques have been possible only in a limited number of systems.

"Fabrication of high-coercive strength magnet materials from the liquid state could lead to a marked reduction in processing steps and hence in cost. The magnets would have theoretical density, the fine particles would be protected from environmental attack, the magnets would have a high degree of particle... [alignment], and the magnets would only require a minimal amount of final

machining...." (1, p. 450)

References/Applicable Publications:

(1) Larson, D. J., Jr.: Zero-G Processing of Magnets. In Apollo-Soyuz Test Project, Summary Science Report, Vol. I, NASA SP-412, pp. 449-470. (post-flight)

(2) Boese, A., McHugh, J., and Seidensticker, R.: Multipurpose Electric Furnace. In Apollo-Soyuz Test Project, Summary Science Report, Vol I, NASA SP-412, pp. 353-365. (post-flight)

(3) Larson, D. J.: Zero-G Processing of Magnets; Experiment MA-070. In Apollo-Soyuz Test Project-Composite MSFC Final Science Report, NASA TM X-73360, January 1977, pp. VI-1 - VI-53. (post-flight)

(4) Prototype Test Report for MA-010 Experiment Cartridges. Tech. Rep. WANL-TME-2867, Westinghouse Electric Corp., Astronuclear Lab., May 1975. (hardware description)

(5) Naumann, R. J. and Mason, E. D.: Zero-G Processing of Magnets. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, p. 62-63. (post-flight)

(6) Larson, D. J. and Pirich, R. G.: Low-G Bridgman Growth of Eutectic MnBi/Bi Magnetic Composites. Fourth American Conference of Crystal Growth, NBS, Gaithersburg, Maryland, July 1978.

(7) Pirich, R. G., Larson, D. J., and Busch, G.: Magnetic and Metallurgical Properties of Directional Solidified Eutectic MnBi/Bi Composites: The Effect of 0-g and Anneal. 24th Conference on Magnetic Materials, Cleveland, Ohio, November 1978.

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Experiment Origin: USA
Mission: STS Launch #18, STS-025 (STS 51-G, Discovery)
Launch Date/Expt. Date: June 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Automated Directional Solidification Furnace (ADSF-1, low temperature version)
Builder of Processing Facility: Originally built for the SPAR program by General Electric (GE) Corporation, Large, Pennsylvania; Refurbished by GE and then NASA Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Directional Solidification of Magnetically Aligned BiMn Alloy (ADSF-1)

This investigation was the second in a series of experiments designed by Larson to study the directional solidification of magnetic composites in the space environment (see Larson, ASTP). (Also related to this research are the experiments of Pirich, SPAR 6 and SPAR 9, and Bethin, SPAR 10 which all can be found in this chapter.)

During the mission, four modules within the Automated Directional Solidification Furnace (ADSF) were employed to process both eutectic and off-eutectic Bi/MnBi magnetic composites. The ADSF was programmed to automatically process the samples over a period of 3 days, largely during astronaut sleep periods. While a furnace temperature of 485 ^{o}C and a thermal gradient of approximately 100 $^{o}C/cm$ was desired for all of the samples, varying furnace translation rates were chosen for each of the modules.

Post-flight analysis of the telemetry record indicated that only one of the furnaces had translated from its starting position. This sample, which was successfully directionally solidified, had a constant furnace translation rate of 0.64 cm/h. "The other samples were melted, held at temperature for long periods, and radially[sic] cooled (furnace cooled)... [F]or each of the cases without translation there is only a record of the furnace "hold" temperature and no record of the sample cooling rate that occurred when the furnaces were cooled. Thus, the samples that did not translate offer only a record of a fluid static geometry and some possible qualitative observations regarding morphology." (2, p. 13). Analysis efforts centered, therefore, on the direc-

tionally solidified, translated sample.

The flight sample was compared to samples processed on Earth. The actual thermal gradient impacted to the translated flight sample was calculated to be 124 $^{o}C/cm$, (higher than the ground-based gradient, 107 $^{o}C/cm$). This higher gradient was attributed to a reduction of convective flow in the low-gravity environment. The STS sample, which had a nonwetting melt/crucible configuration, was found to have increased porosity over ground-processed samples. The sample microstructure exhibited a longitudinal macrosegregation pattern that was "...consistent with theoretical models for diffusion-controlled growth. These data support the conclusion that the thermosolutal convection (lighter solute rejected ahead of the advancing solidification interface), experienced during ground-based solidification, has been effectively damped by the microgravity environment and that diffusion-controlled growth has been achieved.

"However, the region of two-phase, steady-state, diffusion-controlled growth was not isocompositional, as anticipated from theory." (2, p. vi) It was concluded that Soret diffusion was responsible for this macrosegregation. It was noted that increased suppression of the convective currents could be achieved by orienting the sample to reduce orbiter residual gravity contributions.

"Extensive ground-based experiments were conducted attempting to damp the thermosolutal convection or minimize its influences. These included growth orientation relative to gravity, thermal gradient variation (longitudinal and radial), velocity variation, and applied magnetic fields. None have approached diffusion-controlled growth, and the magnetic fields were detrimental, causing increased convection. As a consequence, we have concluded that the flight result is unique." (5, p. vi)

Further discussion of the three non-translating samples was not provided.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Eutectics, Faceted Eutectics, Magnetic Composites, Binary Systems, Two-Phase System, Melt and Solidification, Directional Solidification, Bridgman Technique, Thermal Gradient, Thermal Soak, Furnace Translation, Segregation, Macrosegregation, Wetting, Non-Wetting of Container, Buoyancy Effects, Homogeneity, Thermosolutal Convection, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Diffusion, Soret Diffusion, Diffusion-

Controlled Growth, Multiphase Media, Composition Distribution, Solid/Liquid Interface, Translation Rate, Sample Microstructure, Porosity, Thermal Environment More Extreme Than Predicted, Acceleration Effects, Magnetic Fields, Processing Difficulties, Hardware Malfunction

Number of Samples: four
Sample Materials: bismuth-manganese (Bi/MnBi) (The specific composition of each of these samples was not detailed.) <Note: Although the Principal Investigator reported that Reference (5) listed the specific compositions of each of the samples, these compositions could not be resolved from Reference (5).>
(Bi*Mn*Bi*)
Container Materials: unknown

Experiment/Material Applications:
These investigations and subsequent space experiments hope to "...demonstrate the feasibility of producing improved magnetic composite materials for commercial use. These materials could lead to smaller, lighter, stronger and longer-lasting magnets for electrical motors used in aircraft and guidance systems, surgical instruments and transponders." (1, p. 22.)

References/Applicable Publications:
(1) Space Shuttle Mission 51-G. NASA Press Kit, June 1985, p. 22. (preflight)

(2) Larson, D. J., Jr., Bethin, J., and Dressler, B. S.: Shuttle Mission 51-G, Experiment MPS77F075, Flight Sample Characterization Report. Report RE-753, August 1988, Grumman Corporate Research Center, 43 pp. (post-flight)

(3) Automated Directional Solidification Furnace (ADSF): A Space Shuttle Materials Processing Middeck Payload. Document Developed by Teledyne Brown Engineering under the direction of the Application Payload Projects, Spacelab Payload Projects Office, Marshall Space Flight Center, Huntsville, Alabama. (processing facility and preliminary post-flight experiment results)

(4) Automated Directional Solidification Furnace. In Microgravity Science and Applications Experiment Apparatus and Facilities, document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, pp. 6-7. (processing facility)

(5) Larson, D. J., Jr. and Thompson, B. S.: Off-Eutectic Bi/MnBi Solidification and Soret Transport. Research Report RE- Volume 2, Solid State Physics Research Directorate, Grumman Corporate Research Center, Bethpage, New York, Final Report on Experiment MPS77F075, 41 pp. (post-flight)

(6) Input received from Principal Investigator D. Larson, August 1993.

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Co-Investigator(s): None
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Experiment Origin: USA
Mission: STS Launch #26, STS-26
Launch Date/Expt. Date: September 1988
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment (entire flight package occupied the space of five crew lockers in orbiter middeck)
Processing Facility: Automated Directional Solidification Furnace (ADSF): Four furnace modules each of which processed a single sample. The ADSF was first used aboard sounding rockets, then refurbished for orbiter compatibility including an extended operating time of up to 20 hours.
Builder of Processing Facility: Originally built by General Electric Corporation, Large, Pennsylvania; refurbished by General Electric, NASA Marshall Space Flight Center, Huntsville, Alabama and Teledyne Brown Engineering, Huntsville, Alabama

Experiment:
Directional Solidification of a Magnetic Composite Using the Automated Directional Solidification Furnace (ADSF)

This experiment was the fourth in a series of experiments designed by Larson to investigate the directional solidification of magnetic composites in the space environment (see Larson, ASTP and STS-025 (this chapter), and STS-032 (Chapter 14)).

Four furnaces, (each capable of processing one Mn-Bi magnetic composite sample), were housed in the Automated Directional Solidification Furnace facility. The furnaces were designed to traverse the samples, processing them at a constant melting and solidification speed of 1 cm/h. A processing time of 10.5 hours per sample was expected. Space-produced samples were to be compared to (1) similarly processed Earth-produced samples, (2) earlier processed Shuttle samples (see Larson STS-025 and STS-032) and (3) earlier processed sounding rocket samples (see Pirich SPAR 6, SPAR 9, and Bethin, SPAR 10 (all in this chapter)).

Reportedly, "...only one of the experiment's four furnaces translated, or resolidified, successfully during the ADSF experiment...." (2, p. 1) This furnace "...translated 4.4 cm and then stopped, holding the last portion of the sample to solidify in the thermal gradient for approximately 2.5 h before furnace shutdown. Solidification from this static thermal position would be described as gradient freeze since the thermal gradient extended from the stationary solid-liquid interface beyond the end of the

sample prior to and during cooling." (5, p. 14)

(Reportedly, the non-translated samples were analyzed "...as furnace cooled samples, with gradient freeze and radially cooled regions rather than as directionally solidified samples." (5, p. 14))

<Note: Reference (5) appears to further discuss results related to the translated sample, although specific details of the sample analysis were not clear. Reference (5) stated:>

"The influences of gravitationally-dependent effects on Bridgman-Stockbarger crystal growth of Bi/MnBi eutectics were investigated by conducting microgravity damping experiments on the STS-26 flight of the Space Shuttle 'Columbia', and magnetic field damping experiments at the Grumman Research Center and at the Francis Bitter National Magnet Laboratory, using transverse and longitudinal applied magnetic fields, respectively. These test results were quantitatively compared to undamped one-g baseline results where the gravitational vector was varied relative to the solidification direction. Both microgravity processing and applied magnetic fields were shown to be effective means of damping the natural gravitationally-dependent convection normally encountered terrestrially during Bi/MnBi eutectic solidification. Diffusion-controlled growth during Bridgman-Stockbarger crystal growth was achieved within the Bi/MnBi eutectic in the course of the damped experiments." (5, p. iii)

Please refer to Reference (5) for additional information.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Eutectics, Magnetic Composites, Binary Systems, Melt and Solidification, Directional Solidification, Bridgman Technique, Thermal Gradient, Furnace Translation, Segregation, Buoyancy Effects, Homogeneity, Thermosolutal Convection, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Diffusion, Diffusion-Controlled Growth, Multiphase Media, Composition Distribution, Solid/Liquid Interface, Translation Rate, Sample Microstructure, Magnetic Fields, Hardware Malfunction, Furnace Malfunction, Processing Difficulties

Number of Samples: four
Sample Materials: manganese-bismuth composites (specific compositions unknown)
(Mn*Bi*)

Container Materials: quartz
(Si*O*)

Experiment/Material Applications:
The investigations were "...designed to demonstrate the possibility of producing lighter, stronger, and better-performing magnetic composite materials in a microgravity environment." (1, p. 26)

See also Larson, STS-025.

References/Applicable Publications:
(1) NASA Press Kit: Space Shuttle Mission STS-26. September 1988, pp. 26-28. (preflight)

(2) Investigators Examining Every Particle of Data from STS-26 Experiments. In NASA Marshall Star Newspaper, Vol. 29, Number 16, November 16, 1988, NASA Marshall Space Flight Center, Huntsville, Alabama, p. 1. (post-flight, very short description)

(3) Dumoulin, J. M.: STS-26 Experiment: Automated Directional Solidification Furnace. NASA Fact Sheet, George C. Marshall Space Flight Center, June 1988. (preflight)

(4) Seven Marshall Payloads to Fly on STS-26 in June. Marshall Star, Vol. 28, No. 5, October 7, 1987, pp. 1-2 (preflight; very short description)

(5) Larson, D. J., Jr. and Thompson, B. S.: Bi/MnBi Eutectic Solidification. Research Report RE, Volume 1, Solid State Physics Research Directorate, Grumman Corporate Research Center, Bethpage, New York, Final Report on Experiment MPS77F075, 41 pp. (post-flight)

(6) Input received from Principal Investigator D. Larson, August 1993.

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Experiment Origin: USA
Mission: SPAR 2
Launch Date/Expt. Date: May 1976
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: FWD (Forward) General Purpose Rocket Furnace (GPRF)
Builder of Processing Facility: Unknown

Experiment:
Agglomeration in Immiscible Liquids (74-30/1)

Various mechanisms, which result in the agglomeration of liquid droplets in the host liquid on Earth, are not active in a low-gravity environment:

(1) gravity-dependent Stokes flow (which is the movement of droplets caused by density differences between the liquids) and

(2) gravity-induced convection currents (which result in droplet collisions).

This SPAR 2 experiment was the first in a series of investigations designed by Gelles et al. to study the low-gravity solidification behavior of immiscible liquids. The specific objective of the experiment was to determine the effect of gravity on the structure of two Al-In alloys when cooled through the miscibility gap at a controlled rate.

Prior to the rocket launch, two Al-In samples were prepared: (1) Al-40 wt.% In and (2) Al-70 wt.% In. The pure components of each alloy were placed (In on top of Al, in proper proportion) within alumina crucibles. (Pure components, rather than an alloy, were used "...because greater control of the final alloy composition could be effected in this manner with a system highly prone to macrosegregation." (8, p. 432)) A thermocouple was included in the Al-40 wt.% In system. The two crucibles were enclosed in a stainless steel cartridge and the top cap was welded in place. The cartridge then was subjected to an evacuation/He back-filling process until a final pressure of 1.5×10^{-2} MPa was attained. Testing indicated no He leakage within detectable levels. (See

Reference (8) for further sample preparation details.) The cartridge was placed in one of the three chambers of the SPAR 2 General Purpose Rocket Furnace (GPRF).

Prior to the rocket launch (t = -900 seconds), the samples were heated to about 950 ^{o}C (above the miscibility gap) and held at this temperature up to 154 seconds into the flight (t = + 154 seconds). (Reportedly, a 970 ^{o}C hold temperature was intended, but this temperature discrepancy was not considered important.) At t = +154 seconds, cooling of the samples was initiated by a He gas quench. It was reported that at t = +176, the temperature was 614 ^{o}C and at t = +269 seconds, the temperature was 155 ^{o}C.

Some rocket spin characteristics during the mission were reported. At t = +30 seconds, the rocket was spinning at a rate of 240 rpm about its longitudinal axis; at t = +60 seconds, a rapid despin procedure was initiated.

Ground-based experiments were conducted and the terrestrially processed materials were compared to the flight samples. The post-flight analysis of the two SPAR samples included radiographic and metallographic analysis. The following conclusions from these studies were reported:

(1) Examination of the 1-g processed samples indicated a layered structure with Al-rich regions at the top and In-rich regions at the bottom. A theoretical model to explain the macro- and microstructures of these samples was provided (see Reference (2)).

(2) The type of macrostructure which resulted from low-gravity processing was unexpected: both the 40 wt.% In sample and the 70 wt.% In sample exhibited an annular In region surrounding an Al-rich core. These results were attributed to fluid flow occurring in the low-gravity environment. (An analysis of the possible fluid flow sources within this system was provided.)

(3) Of the fluid flow sources possible in the low-gravity environment, thermocapillary convection and conventional convection were probably active. It was suspected that capillary flow also was active, although this analysis had not yet been completed at the time the available references were published. It was reported that fluid motion due to rocket spin was not significant.

(4) Calculation of the equilibrium configuration of Al and In under low-gravity conditions (based on known surface energy data of the components and estimated interfacial energy data from similar systems) closely agreed with that found for the flight samples and other past results.

(5) Bond number determinations (the ratio of gravity and surface tension forces) indicated that surface tension forces in this alloy system are dominant under low-gravity conditions.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Fluids, Immiscible Alloys, Metals, Binary Systems, Melt and Solidification, Homogeneity, Drops, Droplet Agglomeration, Drop Migration, Stokes Flow, Density Difference, Droplet Collision, Surface Tension, Surface Energy, Interfacial Energy, Buoyancy-Driven Convection, Thermocapillary Convection, Surface Tension-Driven Convection, Marangoni Convection, Capillary Flow, Separation of Components, Phase Separation, Macrosegregation, Solid/Liquid Interface, Liquid/Liquid Interface, Quench Process, Sample Microstructure, Composition Distribution, Acceleration Effects, Rocket Motion, Superconductors

Number of Samples: two
Sample Materials: (1) aluminum-40 wt.% indium and (2) aluminum-70 wt.% indium
(Al*In*)
Container Materials: alumina, Al_2O_3, crucibles contained in stainless steel cartridge
(Al*O*)

Experiment/Material Applications: Several systems exhibit a liquid phase miscibility gap. Some of these materials may be used for applications such as electrical contacts, permanent magnets, or bearings. Potential applications include superconductors, superplastic materials, and catalysts.

References/Applicable Publications:

(1) Gelles, S. H. and Markworth, A. J.: Agglomeration in Immiscible Liquids. In Space Processing Applications Rocket Project, SPAR II Final Report, NASA TM-78125, pp. VI-1 - VI-53, November 1977. (post-flight report)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements, SPAR 1-4)

(3) Agglomeration in Immiscible Liquids at Low Gravity. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, Edited by R. J. Naumann, NASA TM-78217, January 1979, pp. 15-16. (post-flight)

(4) Gelles, S. H. and Markworth, A. J.: Microgravity Studies in the Liquid Phase Immiscible System, Al-In. AIAA Paper 77-122, January 1977.

(5) Moak, D. P., Griesenauer, N. M., and Gelles, S. H.: Undercooling of Materials During Solidification in Space. NASA CR-120750, April 1975.

(6) Gelles, S. H. and Markworth, A. J.: SPAR II Experiment No. 74-30 Agglomeration in Immiscible Liquids. Final Post-Flight Report to NASA Marshall Space Flight Center, December 1976, Battelle Columbus Laboratories, 56 pp. (post-flight)

(7) Gelles, S. H. and Markworth, A. M.: Agglomeration in Immiscible Liquids; Applications of Space Flight in Materials Science and Technology. Proceedings of a Conference Held at the National Bureau of Standards, Gaithersburg, Maryland, April 20-21, 1977, issued September 1978, pp. 25-39. (post-flight)

(8) Gelles, S. H. and Markworth, A. J.: Microgravity Studies in the Liquid-Phase Immiscible System: Aluminum-Indium. AIAA Journal, Vol. 16, No. 5, May 1978, pp. 431-438. (post-flight)

(9) Input received from Principal Investigator A. J. Markworth, June 1993.

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Experiment Origin: USA
Mission: SPAR 5
Launch Date/Expt. Date: September 1978
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: General Purpose Rocket Furnace (GPRF)
Builder of Processing Facility: Unknown

Experiment:
Agglomeration of Immiscible Liquids (74-30)

Following the SPAR 2 experiment concerning the solidification of Al-In alloys (see Gelles, SPAR 2), ground-based studies were conducted on the Al-In system to further investigate the SPAR 2 results and to prepare for the SPAR 5 experiment. These terrestrial studies included (1) spin-up and despin experiments (simulating the spin-up and despin actions of a sounding rocket), (2) Differential Thermal Analysis (DTA) measurements of equilibrium kinetics (to determine the homogenization rate of the liquid Al-In alloys), and (3) Al-In interdiffusion coefficient determinations. Results from these studies coupled with examinations of the SPAR 2 flight samples, indicated that the massive separation observed in the SPAR 2 samples may have been due to the inhomogeneity of the material prior to solidification. It was suspected that this initial concentration gradient contributed to the observed unexpected segregation.

This SPAR 5 experiment was the second in a series of investigations designed by Gelles et al. to study the low-gravity solidification behavior of immiscible liquids (see Gelles, SPAR 2). The specific objectives of the experiment were to (1) determine if the results from the SPAR 2 investigation were due to concentration gradients probably present in the samples prior to the cool down phase and (2) determine if different alloy compositions from those of the SPAR 2 experiment would also show massive separation.

During the SPAR 5 mission, two chambers of the General-Purpose Rocket Furnace (GPRF) were used to process four samples: (1) Al-30 wt.% In, (2) Al-40 wt.% In, (3) Al-70 wt.% In, and (4) Al-90 wt.% In. Samples 2 and 3 were the same compositions processed

during the SPAR 2 experiment. The sample preparation, cartridge design, and experimental procedure were essentially the same as that for the SPAR 2 experiment (see Gelles, SPAR 2) with the following exceptions: (1) a thermocouple was not included in the cartridge containing the 30 and 90 wt.% In alloys, (2) the sample hold temperatures were slightly different (about 980 ^{o}C for SPAR 5 versus 950 ^{o}C for SPAR 2), and (3) the pre-launch times that samples were held above 980 ^{o}C were different (16 hours for SPAR 5 versus 15 minutes for SPAR 2). A thermocouple was included in the capsule containing the 40 and 70 wt.% In alloys.

The SPAR 5 experiment sequence proceeded as follows: after holding the samples at 980 ^{o}C for 16 hours, the rocket was launched (t = 0 seconds). At t = +68 seconds, the rocket despin procedure began. Low-gravity conditions (10^{-3} g) were achieved at t = +84 seconds. At t = +160 seconds cooldown of the samples was initiated. The end of the low-gravity period was estimated to be 329 seconds after launch with (1) solidification of the 40 and 70 wt.% In alloys completed at approximately t = + 392 seconds and (2) solidification of the 30 and 90 wt.% In alloys completed at approximately t = + 352 seconds.

Reportedly, the thermocouple included in the capsule containing the 40 and 70 wt.% alloys "...failed by fracturing at the base of the cartridge during preparation for the flight." (1, p. IV-59) Estimated cooling rates were 10.0 ^{o}C/sec for the 40 and 70 wt.% In samples and 10.6 ^{o}C/sec for the 30 and 90 wt.% In samples. "It should be noted... that the cooling rates experienced during SPAR [5] were somewhat lower than desired... ([approximately] 10 C/second vs. the goal of 14 C/second).... [T]his factor has led to the likelihood that the indium-rich phase was still liquid at the end of the low-g (<1 x 10^{-3}g) period. Although this factor is not expected to alter the general conclusions of the study, it does introduce some uncertainty in the results. The absence of a thermocouple internal to the melt adds further to the degree of uncertainty in the results." (1, p. IV-62)

Post-flight examination of the flight samples included radiography and optical microscopy studies. The results from the 40 and 70 wt.% In alloys were similar to those from the SPAR 2 experiment: the flight samples consisted of a macroscopically sized Al-rich core surrounded by an In-rich alloy. Samples processed on Earth consisted of a layered structure. Subtle differences between the SPAR 2 and SPAR 5 samples were attributed to the different cooling rates. The structure of the low-gravity processed, 30 wt.% In alloy was very similar to the 40 wt.% In flight alloy "...and thus provided little further understanding of the phase separation process. The 90 weight percent alloy, however, did provide some new insight into the mechanisms that may be contributing to massive phase separation. Most notable

among the observations made on the alloy is the presence of an annular zone denuded of aluminum-rich spheres around the massively separated aluminum-rich core. This observation, coupled with the fact that there is an increasing concentration of aluminum-rich spheres close to the central core, has provided evidence supporting the theory that the aluminum-rich spheres have migrated from the outer regions of the alloy into the interior, presumably under the action of surface tension gradients." (1, p. IV-9) These results, as well as those from the SPAR 2 experiment, led to the following conclusions:

(1) Massive separation in the flight samples was not due to a lack of homogeneity in the melt at the beginning of the cooling period.

(2) It was likely that surface tension driven flows were active and were significant in forming the observed structures in both the SPAR 2 and SPAR 5 samples (see Reference (1) for further details).

(3) It was highly probable that migration of Al-rich droplets in the In-rich flight samples was due to thermocapillary migration. This was evidenced by particle coalescence, a process which reduces the probability of particle pushing by the solidification front.

(4) No microstructural or macrostructural effects could be attributed to the failure of the samples to solidify prior to the end of the low-gravity period.

(5) "Ground-based experiments conducted on rapidly cooled aluminum-indium alloys have shown the phase separation to be extremely sensitive to composition with regions near the critical composition providing massively separated phases which nucleated and grew in much less than a second." (1, p. IV-100) This instability was attributed to the (1) high volume fraction of the second phase and (2) close proximity of neighboring droplets.

(6) There was evidence that in the Al-rich region of the miscibility gap, the In-rich phase nucleated at the crucible wall during cooling. This nucleation was attributed to the wetting properties of the materials. As the In-rich content increases, the tendency for Al-rich droplets to nucleate at the crucible wall increases.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Fluids, Immiscible Alloys, Metals, Binary Systems, Melt and Solidification, Cooling Rate, Sample Homogeneity, Drops, Droplet Agglomeration, Drop Coalescence, Drop Migration, Density Difference, Surface Tension, Surface Energy, Interfacial Energy, Free Surface, Buoyancy-Driven Convection, Thermocapillary Convection, Surface Tension-Driven Convection, Marangoni Convection, Marangoni Movement of Droplets, Capillary Flow, Diffusion, Interdiffusion, Diffusion Coefficient, Separation of Components, Segregation, Phase Separation, Solutal Gradients, Surface Tension Gradients, Solid/Liquid Interface, Liquid/Liquid Interface, Quench Process, Sample Microstructure, Composition Distribution, Precipitation, Nucleation, Nucleation Sites, Wetting, Wetting of Container, Crucible Effects, Material Interaction with Containment Facility, Rocket Motion, Vehicle Re-Entry Forces/Vibration, Hardware Malfunction, Incomplete Sample Processing, Superconductors

Number of Samples: four
Sample Materials: aluminum-indium alloy with 30, 40, 70 and 90 wt.% indium
(Al*In*)
Container Materials: alumina, Al_2O_3, crucibles contained in stainless steel cartridge
(Al*O*)

Experiment/Material Applications:
See Gelles, SPAR 2

The 30 wt.% In alloy was employed in this experiment to determine the effect of a smaller In concentration on the tendency toward massive separation. The 90 wt.% In alloy was selected because (1) the precipitating phase has a low concentration and (2) the tendency of Al droplets to precipitate at the crucible walls (analogous to In precipitation in high-Al concentration alloys) could be investigated.

References/Applicable Publications:
(1) Gelles, S. H. and Markworth, A. J.: SPAR V Experiment No. 74-30 Agglomeration in Immiscible Liquids. In Space Processing Application Rocket Project, SPAR V Final Report, August 1980, NASA TM-78275, pp. IV-i - IV-105. (post-flight)

(2) Gelles, S. H. and Markworth, A. J.: Low Gravity Experiments on Liquid Phase Miscibility Gap (LPMG) Alloys-Materials Experiment Assembly. In Proceedings of the 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, June 1983, pp. 307-312. (SPAR and STS experimentation)

(3) Agglomeration in Immiscible Liquids at Low Gravity. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, Edited by R. J. Naumann, NASA TM-78217, pp. 15-16, January 1987. (post-flight)

(4) Input received from Principal Investigator A. J. Markworth, June 1993.

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Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Payload Bay, OSTA-2 Payload Pallet Platform, Materials Experiment Assembly (MEA-A1)
Processing Facility: Gradient and isothermal versions of the SPAR General Purpose Rocket Furnace (GPRF)
Builder of Processing Facility: Unknown

Experiment:
Liquid Phase Miscibility Gap: (1) Gradient Cooling Experiment and (2) Isothermal Plunger Experiment

This STS-007 space shuttle experiment was the third in a series of investigations designed by Gelles et al. to study the low-gravity solidification behavior of immiscible liquids (see Gelles, SPAR 2, SPAR 5).

<Note: A document published after the return of STS-007 reported that, during the mission, three experiments were performed using the GPRF: (1) the gradient solidification of an Al-In alloy, (2) the isothermal solidification of a Te-Tl alloy, and (3) the isothermal solidification of an Al-In alloy. However, no discussion of the objectives or results of the Te-Tl experiment could be located in published literature at this time. Therefore, only the two Al-In experiments are summarized below.>

Two experiments were discussed in detail in the available publications: (1) the gradient solidification of an Al-90 wt.% In alloy with thermal gradient and (2) the isothermal solidification of an Al-90 wt.% In alloy with a plunger system.

Experiment #1: Solidification of Al-In with Thermal Gradient

The specific objective of this experiment was to investigate (1) droplet migration driven by surface tension gradients and (2) particle pushing by a moving solidification interface.

Prior to the STS launch, plugs of the alloy components in elemental form were contained in an alumina crucible. The specimen was equipped with two thermocouples, one near the top of the sample

material and the other near the bottom. A copper chill block was placed at the bottom of the alumina crucible for heat extraction. This entire assembly was sealed in a stainless steel cartridge which was evacuated and back filled with He (0.0173 MPa). The cartridge was configured within the Spacelab General Purpose Rocket Furnace (G-GPRF).

During the experiment, the sample was (1) heated to 974 ^{o}C, (2) held at this temperature for 8.1 hours, and (3) cooled at a rate of 0.28 $^{o}C/sec$ (with an average gradient of 8.1 $^{o}C/cm$). It was expected that this procedure would result in (1) a single phase liquid which would undergo phase separation with L_I droplets precipitating in the L_{II} host liquid upon cooling, and (2) migration of the droplets to the warmer regions (Marangoni driven migration). A computer simulation of this process had predicted this behavior (see Reference (4)).

Post-flight, the STS-007 sample was compared to (1) samples processed on Earth, using the nearly the same thermal conditions and (2) the Al-90 wt.% In sample processed during the SPAR 5 sounding rocket experiment. It was noted that comparison was complicated because (1) the SPAR 5 sample was subjected to a cooling rate through the miscibility gap approximately 100 times faster than the isothermal samples from the STS-007 experiment and (2) the SPAR 5 samples did not completely fill the crucible and a free surface resulted while the material was molten.

Optical and electron microscopic methods were used to evaluate the STS gradient sample. It was reported that, contrary to expectations, the L_I droplets "...appeared to be concentrating and coalescing mainly at the cooler end of the sample.... In contrast, the dendrites which form below the monotectic temperature were found to be uniformly distributed in the flight sample." (1, p. 418) A shrinkage cavity was also located in the middle of the sample as well as an expected cavity at the hot end. Several possible explanations of the L_I droplet behavior were reported (see Reference (1) for a complete discussion). The most likely reasons for this behavior were reported to be (1) the non-uniform temperature gradient, (2) an incorrectly assumed behavior of the interfacial energy between L_I and L_{II}, and (3) the solutocapillary effects.

Experiment #2: Solidification of Al-In with a Plunger System (Isothermal Experiment)

The specific objective of this experiment was to (1) determine if during phase separation, surface tension-driven convection currents (which originate at a free surface) contribute to droplet coalescence and (2) determine if the Liquid 1 (L_I) droplets which

form during the phase separation process migrate due to a thermal gradient.

Prior to the STS launch, plugs of the alloy components in elemental form were contained in an alumina crucible. A close-fitting alumina plunger was placed in the top part of the crucible. The plunger contact with the sample material was maintained with a quartz spring in a stainless steel retainer. The purpose of the plunger was to avoid the presence of a liquid free surface (which would result in surface tension-driven fluid flow). A thermocouple was included at the bottom of the crucible. This entire arrangement was sealed in a stainless steel cartridge which was evacuated and back-filled with a partial pressure of He designed to reach 0.1 MPa at the intended hold temperature 970 ^{o}C. Two of these cartridges (which were duplicates, designated as P-10 and P-2-10) were configured in the isothermal version of the GPRF.

Post-flight examination of the isothermal samples and subsequent comparison of the samples to ground-based-produced samples and the Al-90 wt.% In SPAR 5 sample were similar to the post-flight examination of the STS-007 gradient sample. "It should be noted that in the case of Cartridge P-10, molten metal attack of the thermocouple has occurred causing some errors in the temperature readings." (1, p. 420) The actual temperature readings recorded were between 984 ^{o}C and 916 ^{o}C. "The actual temperatures are suspected to be higher than the indicated temperatures...." (1, p. 420)

It was reported that both flight samples contained few relatively large L_I droplets with many smaller ones distributed through the In-rich matrix. These smaller droplets were adjacent to, but generally not touching, the crucible wall. "The L_I drops in these samples appear to be somewhat more finely dispersed than in the free surface SPAR [5] sample...." (1, p. 420) This result led "...to the tentative conclusion that the surface tension driven convection currents originating from a free surface[,]... to a small extent[,] contribute to the coalescence of the L_I drops. It is clear however that another mechanism must be responsible for most of the observed coalescence. The one most suspected is droplet migration arising from gradients in surface tension." (1, p. 421) The ground-processed sample consisted of an Al-rich layer above an In-rich layer. The dendritic structure in the isothermal flight samples was similar to the gradient sample. However, in the ground-processed isothermal sample, the dendrites were driven by buoyancy forces to the top of the In-rich layer.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Fluids, Immiscible Alloys, Metals, Metallic Matrix, Binary Systems, Melt and Solidification, Directional Solidification, Cooling Rate, Sample Homogeneity, Drops, Drop Coalescence, Drop Migration, Thermomigration, Marangoni Movement of Droplets, Droplet Dispersion, Density Difference, Surface Tension, Surface Energy, Interfacial Energy, Buoyancy-Driven Convection, Buoyancy Forces, Free Surface, Free Surface Elimination, Piston System, Thermocapillary Convection, Surface Tension-Driven Convection, Marangoni Convection, Thermosolutal Convection, Capillary Flow, Separation of Components, Segregation, Phase Separation, Solutal Gradients, Surface Tension Gradients, Thermal Gradient, Isothermal Processing, Thermal Soak, Solid/Liquid Interface, Liquid/Liquid Interface, Solidification Front Physics, Interface Physics, Sample Microstructure, Composition Distribution, Precipitation of Second Phase, Dendrites, Cavity, Sample Shrinkage, Superconductors, Hardware Malfunction, Thermal Environment More Extreme Than Predicted

Number of Samples: three
Sample Materials: aluminum-indium alloys, Al-90 wt.% In; a tellurium-thallium alloy
(Al*In*, Te*Tl*)
Container Materials: Alumina (Al_2O_3) crucible within a stainless steel cartridge
(Al*O*)

Experiment/Material Applications:
See Gelles, SPAR 5.

References/Applicable Publications:
(1) Gelles, S. H. and Markworth, A. J.: Space Shuttle Experiments on Al-In Liquid Phase Miscibility Gap (LPMG) Alloys. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 417-422. (post-flight)

(2) Gelles, S. H. and Markworth, A. J.: Low-Gravity Experiments On Liquid Phase Miscibility Gap (LPMG) Alloys: Materials Experiments Assembly (MEA). In ESA 4th European Symposium on Material Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, pp. 307-312. (preflight)

(3) Harris, E. G.: Materials Experiment Assembly (MEA) Acceleration Summary, STS-7, Marshall Space Flight Center, JA62-004, July 1984.

(4) Gelles, S. H.: Liquid Phase Miscibility Gap Alloys - MEA A1 Experiments. Final post flight report to NASA-MSFC on Contract NAS8-32952. <Note: The date this report was published is unclear.>

(5) General Purpose Rocket Furnace. In Microgravity Science and Applications Experiment Apparatus and Facilities document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, pp. 3-4. (processing facility)

(6) Naumann, R. J.: Microgravity Science and Applications. In: In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 23-24. (post-flight)

(7) Input received from Principal Investigator A. J. Markworth, June 1993.

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Experiment Origin: USA
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Payload Bay Materials Experiment Assembly (MEA-A2)
Processing Facility: Isothermal General Purpose Rocket Furnace (I-GPRF)
Builder of Processing Facility: Unknown

Experiment:
Liquid Phase Miscibility Gap Materials

<Note: Publications which detailed the actual D1 experimental setup or post-flight results of this investigation could not be located at this time. The following summary was based on References (1) and (4), which were published prior to the Spacelab D1 mission.>

This Spacelab D1 experiment was the fourth in a series of investigations designed by Gelles et al. to study the low-gravity solidification behavior of immiscible liquids (see Gelles, SPAR 2, SPAR 5, STS-007). The specific objective of this experiment was to examine the space-produced microstructure of an Al-In alloy and determine the effect of (1) the minimization of gravity, (2) the minimization of surface tension-driven convection currents (using a crucible system with a plunger), and (3) the reduction of the interaction between droplets and crucible wall material.

During the Spacelab D1 mission, the General Purpose Rocket Furnace was to be used to process an Al-40 wt.% In alloy. The alloy was to be contained in a crucible which also held a plunger to prevent the formation of a free surface (gas/liquid interface) during sample processing. It was anticipated that the crucible would be heated to 970 (+/- 10) ^{o}C at a rate which would avoid thermal shock to the ceramic components (>15 minutes). The cartridge was to be held at this temperature, for 12 hours and then cooled at a rate of 6.0 (+/- 0.5) ^{o}C to below 600 ^{o}C. Cooling from 600 ^{o}C to below 100 ^{o}C was expected to occur at a rate near 6 ^{o}C/min.

No post-flight discussion of the actual experimental procedure or results from this experiment could be located at this time.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Fluids, Immiscible Alloys, Metals, Binary Systems, Melt and Solidification, Homogeneity, Drops, Drop Coalescence, Drop Migration, Thermomigration, Marangoni Movement of Droplets, Density Difference, Surface Tension, Surface Energy, Interfacial Energy, Buoyancy-Driven Convection, Free Surface, Free Surface Elimination, Thermocapillary Convection, Surface Tension-Driven Convection, Separation of Components, Segregation, Phase Separation, Solutal Gradients, Surface Tension Gradients, Thermal Soak, Solid/Liquid Interface, Liquid/Liquid Interface, Sample Microstructure, Composition Distribution, Precipitation of Second Phase, Crucible Effects, Material Interaction with Containment Facility

Number of Samples: one
Sample Materials: <Note: The composition of the actual flight sample was not reported in the available documents. A document published prior to the flight indicated that the sample was to be Al-40 wt.% In>
(Al*In*)
Container Materials: unknown

Experiment/Material Applications:
See Gelles, SPAR 5.

References/Applicable Publications:

(1) Gelles, S. H.: Liquid Phase Miscibility Gap Materials. In Scientific Goals of the German Spacelab Mission D1, German Publication, WPF, p. 143. (preflight)

(2) Materials Processing Experiments in Space: MEA-A2 Payload. Brochure available from Application Payload Projects NASA/MSFC, Huntsville, Alabama. (MEA; preflight)

(3) General Purpose Rocket Furnace. In Microgravity Science and Applications Experiment Apparatus and Facilities, document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, pp. 4-5. (processing facility)

(4) Naumann, R. J.: Microgravity Science and Applications. In: In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 23-24. (appears to be discussing results of this flight)

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Experiment Origin: Federal Republic of Germany
Mission: SPAR 2
Launch Date/Expt. Date: May 1976
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: FWD (Forward) General Purpose Rocket Furnace (silicon carbide tube furnace)
Builder of Processing Facility: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Solidification Behavior of Al-In Immiscible Alloys Under Zero-Gravity Conditions (74-62) (SOLUOG)

"Melts of an alloy system with [a] miscibility gap in the liquid state, when homogeneous at high temperatures, should exhibit different separation mechanisms when entering the miscibility gap during the cooling process, depending on whether separation starts within or outside the range of spinodal decomposition. Even alloys with a noncritical composition are capable of spinodal separation, if the binodal temperature is undercooled to the spinodal temperature. The spinodal separation is characterized by spontaneous decomposition of the melt without any nucleation leading to extremely fine and uniform dispersion of the two phases. Under zero-g conditions and at high cooling rates, there should be neither a segregation of the two phases nor an essential coalescence of the droplets of the two melts...." (1, p. VIII-3)

This SPAR 2 experiment was the first in a series of investigations designed by Löhberg and/or Ahlborn et al. to study the solidification of metallic alloys under low-gravity conditions. The specific objective of the experiment was to investigate the decomposition and crystallization behavior of two immiscible alloys which are characterized by a miscibility gap in the liquid state: (1) 60 at.% Al-40 at.% In (sample 60/40) and (2) 89 at.% Al-11 at.% In (sample 89/11). (The chosen alloys had a critical temperature of approximately 830 ^{o}C.)

The experiment was "...expected to provide an answer to the question whether alloys of different compositions exhibit different structures depending on the separation mechanism." (1, p. VIII-3) It was anticipated that the 60/40 sample would decompose spinodally and that the 89/11 sample would decompose by nucleation. The final structures of the samples were expected to exhibit a uniform dispersion of the In-rich and Al-rich phases, although each sample was expected to contain a different size and arrangement of second phases.

Prior to the flight experiment, several ground-based studies were performed to (1) determine if the available phase diagram was sufficiently accurate concerning critical composition and temperature, (2) determine the required holding time above the critical temperature to assure complete homogenization of the melts, (3) select the crucible material and crucible sealing material suitable for these alloys, and (4) select metallographic examination methods which avoid unnecessary material loss. The ground-based samples were processed using various cooling rates for comparison to the flight specimens. (Further details of these preflight experiments are included in Reference (1).)

The two immiscible samples for the flight experiment were composed of pure Al and In plates (the In configured on top of the Al). Each sample was contained in a sintered alumina crucible which was evacuated and sealed with a cementing material. Both crucibles were placed in a high-temperature resistant ferritic steel (Thermax 4742) cartridge which was evacuated and welded air-tight. The cartridge was inserted into the SPAR General Purpose Rocket Furnace (GPRF).

Just prior to the rocket launch, the recorded temperature (based on thermocouples located on the outside of the cartride) was 980 ^{o}C. After launch, the temperature rapidly fell to 950 ^{o}C and remained at that point for about 120 seconds. Cooling was initiated at 150 seconds after launch by introducing helium into the experiment chamber. At about 300 seconds after launch, the temperature was 150 ^{o}C. Cooling rates decreased from 17.5 K/sec to 1 K/sec as the sample temperatures decreased from 950 ^{o}C to 150 ^{o}C.

Comparison of this temperature data to that from another experiment, which was also performed in the GPRF (see Gelles, SPAR 2), led to the conclusion that the monotectic reaction occurred isothermally.

Post-flight examination of the two Al-In samples revealed that the expected phase arrangement was not achieved. However, the stratification observed in the ground-processed samples also was not seen. "In the [low-gravity processed] alloy 60/40, an Al-

rich globule was observed, whose interior was filled with Al-containing In and which was enveloped by Al-containing In. The Al globule has an interconnection point. In the second sample [sample 89/11] the In-rich component accumlated in the upper part of the melt regulus, but not in a stratified manner. Here too, is a narrow In-rich zone at the whole regulus surface. The Al crystallizing at the monotectic temperature (636 ^{o}C) has an equiaxial form and the Al crystallizing below this temperature has a fine dendritic form. [See Reference (1) for micrographs of the sample.]

"In the [low-gravity processed] Al-rich sample 89/11, a sphere could not be formed for geometrical reasons, but a tendency towards sphere formation is obvious. Consequently, the solidification occurred essentially towards the heat flow directed to the bottom, with the In-rich melt being displaced towards the top. In the sample 60/40, by contrast, the following process should have taken place. The spherical Al-rich melt was surrounded by an In-rich melt when the crystallization started at the monotectic temperature and advanced radially to the center against the heat flow going regularly into all directions. In this process, the larger amount of the separated In-rich melt was displaced into the interior of the sphere and was subjected (because of the strong volume contraction during solidification of the aluminum) to a rising pressure which finally led to the Al sphere breaking through a weak point. The resulting current through the 'channel' can still be traced in the solidified sample by 'hollow vortices' existing only at this point.

"The fact that the melt reguli are surrounded by In-rich melt may be understood from the different interface and surface energies of the In-rich and Al-rich melts. Accumulation of In in the sample surfaces as well as in the interface between melt and aluminum oxide crucible may be the result of the lower surface and interface energies of the In-rich melt." (1, p. VIII-42)

It was reported that important questions, still remained which could be answered with both short-term and long-term, low-gravity experiments:

(1) Does a critical growth rate exist below which displacement of the second phase (In-rich melt) by the crystallizing phase (Al-rich melt) does not occur? (Reportedly, experiments in which the crystallization rate was varied would help answer this question.)

(2) What are the effects of surface and interfacial energies on the separation and crystallization of phases? (Reportedly, experiments in which the crucible material and/or the sample length/diameter ratios were varied would help answer this question.)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Metals, Metallic Matrix, Binary Systems, Two-Phase System, Monotectic Compositions, Isothermal Processing, Melt and Solidification, Sample Homogeneity, Drops, Sphericity, Drop Coalescence, Droplet Dispersion, Density Difference, Surface Tension, Surface Energy, Interfacial Energy, Separation of Components, Segregation, Phase Separation, Spinodal Decomposition, Solid/Liquid Interface, Liquid/Liquid Interface, Undercooling, Growth Rate, Cooling Rate, Sample Microstructure, Composition Distribution, Nucleation, Dendritic Structure, Quench Process, Aspect Ratio, Crucible Effects, Material Interaction with Containment Facility

Number of Samples: two
Sample Materials: (1) Al-40 at.% In and (2) Al-11 at.% In (Al*In*)
Container Materials: alumina, Al_2O_3, contained in a high-temperature resistant, ferritic steel Thermax 4742 cartridge (Al*O*)

Experiment/Material Applications:
The Al-In alloys were selected for this experiment because (1) there is a large difference in density between the Al-rich and In-rich melts and (2) the critical temperature of the miscibility gap (approximately 830 ^{o}C) was low enough to allow homogenization of the melts using the available sounding rocket hardware.

References/Applicable Publications:
(1) Ahlborn, H.: Segregation and Solidification of Liquid Aluminum-Indium Alloys Under Zero Gravity Conditions. In Space Applications Rocket Project, SPAR 2- Final Report, NASA TM-78125, pp. VIII1-VIII44, November 1977. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement of Accleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements, SPAR 1-4)

(3) Ahlborn, H. and Löhberg, K.: Aluminum Indium Experiment SOLUOG--A Sounding Rocket Experiment on Immiscible Alloys. 17th Aerospace Sciences Meeting, New Orleans, Louisiana, January 15-17, 1979, 4 pp.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 1
Launch Date/Expt. Date: December 1977
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1 (Isothermal four-chamber furnace)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Germany

Experiment:
Al-Pb Dispersion

Aluminum alloys containing lead dispersions are expected to possess excellent properties for bearing applications. Because of large density differences of the alloy constituents, however, homogeneous Al-Pb alloys cannot be obtained on Earth by conventional casting techniques. It was expected that in a reduced gravity environment, (1) gravity-driven sedimentation of the Pb in the Al melt would be reduced, (2) a fine liquid-liquid dispersion would be obtained, and (3) the fine dispersion could be conserved in the solid by rapid cooling.

This TEXUS 1 experiment was designed to study the low-gravity solidification of an immiscible alloy. The specific objective of the experiment was to produce an Al-Pb alloy with a homogeneous dispersion of Pb in an Al matrix.

During the mission one chamber of the isothermal four-chamber furnace module was used to melt and resolidify a compact powder sample. The Al sample contained 8 wt.% Pb, 3.5 wt.% Si, 1.5 wt.% Cu, and 1 wt.% Sn. While under low-gravity conditions, the temperature of the material was (1) raised above the miscibility gap (1100 ^{o}C) and (2) then lowered below 1000 ^{o}C (to form the required fine, liquid-liquid dispersion). The material was rapidly cooled before the low-gravity period ended.

Post-flight analysis of the sample revealed the formation of pores which was attributed to insufficient densification and/or degassing during sample preparation. Also present were oxide layers which surrounded the lead particles. (These layers were also present in a sample processed terrestrially.) Reportedly, the oxide layers prevented the formation of a homogeneous melt.

Thus, the low-gravity and 1-g samples had similar features (although the size of the Pb particles in the flight sample was smaller than in the 1-g material).

No further information concerning this experiment could be located at this time.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Metals, Metallic Matrix, Powder Metallurgy, Melt and Solidification, Isothermal Processing, Cooling Rate, Casting, Phase Separation, Homogeneous Dispersion, Droplet Dispersion, Particle Dispersion, Liquid/Liquid Dispersions, Particle Size Distribution, Density Difference, Separation of Components, Sedimentation, Segregation, Solid/Liquid Interface, Liquid/Liquid Interface, Bubble Formation, Bubble Removal, Sample Microstructure, Pores, Oxide Layer, Thin Films, Quench Process

Number of Samples: one
Sample Materials: single aluminum sample comprised of 8 wt.% Pb, 3.5 wt.% Si, 1.5 wt.% Cu, 1 wt.% Sn
(Al*Pb*Si*Cu*Sn*)
Container Materials: Si_3N_4 (crucible sealed at normal atmosphere)
(Si*N*)

Experiment/Material Applications:
See Experiment section above.

References/Applicable Publications:
(1) Hodes, E. and Steeg, M.: Production of an Aluminum Lead Alloy in Microgravity. Z. Flugwiss. Weltraumforsch.2 (1978), Heft 5, pp. 337-341. (in German; English abstract)

(2) Final Report, TEXUS-I, DFVLR-BPT, 1978.

(3) Input received from Experiment Investigator, August 1988 and July 1993.

(4) Al-Pb Bearing Alloy. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 246-247. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 1
Launch Date/Expt Date: December 1977
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: isothermal furnace. (One of four available chambers within the furnace was employed for this experiment.)
Builder of Processing Facility: ERNO, Raumfahrttechnik GmbH, Bremen, Germany

Experiment:
<u>Stability of Compound Mixtures/Powder Metallurgy (Composite Materials I: Liquid-Liquid-Gas Systems)</u>

<Note: Walter performed two experiments on TEXUS 1 which involved the TEM 01-1 experiment module. Details of the other experiment can be found in Chapter 5: "Composites with Solid Particles.">
This TEXUS 1 experiment was the first in a series of investigations designed by Walter et al. to explore the low-gravity stability of multicomponent liquid-liquid systems during melting, thermal soak, and solidification. The specific objective of the experiment was to examine the mechanisms (and their relative importance) which drive component separation. These mechanisms not only include (1) sedimentation and buoyancy (gravity effects), but also (2) volume changes, (3) interparticle forces, (4) the motion of droplets in a temperature gradient, (5) interaction of liquid and gaseous inclusions with an advancing solidification front, (6) wetting, (7) liquid spreading, and (8) coalescence. Low-gravity processing permitted closer examination of mechanisms often masked by overwhelming gravity effects.

A composite-powder model system representing a liquid-liquid-gas mixture was chosen for the TEXUS 1 flight. The sample, identified as sample I(5), consisted of Ag particles (32 to 50 micron grain size) and irregularly shaped Na-glass particles. The sample had an Ag/Na-glass volume ratio of 4:1 and was (1) compressed to a residual pore volume of 10% and (2) annealed (on Earth) in hydrogen at 500 $^{\circ}$C and 1 atmosphere for 30 minutes. (Silver (Ag) was chosen as the matrix material and the glass particles were statistically distributed.)

The sample, which was placed in a molybdenum crucible (TZM alloy), was housed with four other samples in a single stainless steel cartridge. <Note: These four other samples consisted of a solid-liquid-gas system and are detailed in Chapter 5, "Composites with Solid Particles" (see Walter, TEXUS 1).>

The samples were processed in one of the four chambers of the TEM-01 isothermal furnace. Reportedly, the cartridge was preheated to 850 °C prior to lift-off and held at this temperature during launch. Once microgravity conditions had been achieved ($<10^{-4}$ g), the samples were heated to 1100 °C. After 30 seconds at this temperature, the samples were cooled such that they were below 600 °C by rocket re-entry.

Analysis of a similar Ag/Na-glass sample, remelted under 1-g conditions, illustrated a significant degree of separation because of the large density differences between the compound elements. It was reported that there was also an unexpected amount of separation in the flight Ag/Na-glass sample. Sample I(5) consisted of "...a large drop of Ag without any inclusion or pores and a single pore... surrounded by a layer of glass." (2, p. 32)

Sample I(5) had a low surface free energy configuration. This result was highly significant. It indicated that the driving forces for separation of such components in the liquid state were not restricted to buoyancy and sedimentation. It was also apparent that rearrangement of the components occurred very rapidly since melting, soak, and resolidification occurred within the 5-minute low-gravity period.

Reportedly, the interfacial energy between Ag-melt and glass was calculated to be about 900 mN/M and the contact angle was about 90° at 1000 °C (Ag melt temp. = 960.8 °C). However, the contact angle between the glass and molybdenum (crucible wall) was less than or equal to 0°. Therefore, the most stable configuration was a layer of glass surrounding a silver core. In order to determine which mechanism was responsible for this configuration (motion of droplets due to a thermal gradient or chain formation) two samples having a larger percentage of glass were processed on TEXUS 2 (see Walter, TEXUS 2, samples II(4) and II(5)).

It was later reported that "These results indicated that separation can take place under microgravity conditions as well and can be driven by surface tension gradients. The preparation of heterogeneous alloys or composites from powder mixtures is, therefore, not possible with mixtures having non-wetting components and pore volume or with degassing materials." (4, p. 236)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Immiscible Fluids, Melt and Solidification,Isothermal Processing, Annealing, Powder Metallurgy, Metallic Matrix, Glasses, Multiphase Media, Model Materials, Binary Systems, Phase Separation, Separation of Components, Stability of Dispersions, Liquid/Liquid Dispersions, Solid/Liquid/Gas Dispersion, Density Difference, Drops, Drop Migration, Marangoni Movement of Droplets, Thermomigration, Sedimentation, Buoyancy Effects, Wetting, Wetting of Container, Contact Angle, Surface Energy, Interfacial Energy, Surface Tension Gradients, Liquid Spreading, Drop Coalescence, Pores, Inclusions, Thermal Soak, Solidification Front Physics, Inclusion and/or Rejection of Particles, Liquid/Liquid Interface, Liquid/Gas Interface, Solid/Liquid Interface, Volume Change, Liquid Phase Sintering

Number of Samples: one
Sample Materials: Ag particles (32 to 50 micron grain size) and irregularly shaped Na-glass particles
(Ag*Na*)
Container Materials: Molybdenum crucible (TZM alloy) with outer stainless steel envelope
(Mo*)

Experiment/Material Applications:

Earlier low-gravity experiments indicated that sedimentation and buoyancy were not the only forces separating components of composite materials (see, for example, Kawada, Skylab SL-3 and SL-4 (Chapter 5); Uhlman, SPAR missions (Chapter 5)). Other mechanisms (as detailed in the above experiment summary) could contribute to separation. It was necessary, therefore, to determine the extent and relevance of each of these forces.

The materials used in both Walter's TEXUS 1 experiments (one liquid-liquid-gas sample (Ag-Na glass) and four solid-liquid-gas samples) were chosen for many reasons: (1) the melt temperatures of each were below that of the available maximum furnace temperature, (2) large density differences between materials could illustrate separation due to residual acceleration components, and (3) wetting and non-wetting powder combinations would indicate separation due to this material characteristic. In addition, (1) no chemical reaction or solubility of components would occur, (2) plasticity of matrix material (Ag) allowed density control, (3) control of surface contamination and oxidation was possible, (4) the vapor pressure at maximum temperature would be low, and (5) the powders were available in the required form (particle

shape, etc.).

References/Applicable Publications:

(1) Walter, H. U.: Stability of Multicomponent Mixtures Under Microgravity Conditions. In Proceedings of the 3rd European Symposium on Material Sciences in Space, Grenoble, April 24-27, 1979, ESA SP-142, p. 245. (post-flight)

(2) Walter, H. U. and Ziegler, G.: Stability of Multicomponent Mixtures. In Shuttle/Spacelab Utilization Final Report, Project TEXUS, 1978, Technological Experiments in Micro-gravity, pp. 27-47. (TEXUS 1 and 2; this paper is the same as the Grenoble paper above)

(3) Walter, H. U. and Ziegler, G.: Rearrangement and Separation Processes During Liquid Phase Sintering Under Microgravity Conditions. In Proceedings of the European Sounding-Rocket, Balloon and Related Research with Emphasis on Experiments at High Latitudes, Ajaccio, Corsica, April 24-29, 1978, ESA SP-135, pp. 345-352. (post-flight)

(4) Composite Materials I. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 236-237. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 2
Launch Date/Expt Date: November 1978
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1
Builder of Processing Facility: ERNO, Raumfahrttechnik GmbH, Bremen, Germany

Experiment:
Stability of Compound Mixtures/Powder Metallurgy (Composite Materials II: Liquid-Liquid Systems)

<Note: Walter performed two experiments on TEXUS 2 which involved the TEXUS TEM 01-1 experiment module. Details of the other experiment can be found in Chapter 5 "Composites with Solid Particles."> This TEXUS 2 experiment was the second in a series of investigations designed by Walter et al. to explore the low-gravity stability of multicomponent liquid-liquid systems during melting, thermal soak, and solidification (see Walter, TEXUS 1). The specific objective of the experiment was to examine the mechanisms (and their relative importance) which drive component separation.

Two-component systems (representative of liquid-liquid systems) were examined. Reportedly, because the chosen samples had no pores or free surfaces "...[(1)] Marangoni-flow generated at liquid-gas interfaces, [(2)] flow induced by volume expansion of gaseous inclusions, [(3)] melt bridge formation and resulting forces on particles, and [(4)] especially capillarity effects and coalescence..." (2, p. 36) should not be significant when resolving separation mechanisms. (All of these factors influenced the results from the TEXUS 1 experiment (see Walter, TEXUS 1).)

Two powder samples (prepared to the theoretical density) were selected for study. The first sample (designated as Sample II(4)), consisted of Ag (particle diameter of 100 to 200 microns) and Na-glass (particle diameter of 100-160 microns). The sample had an Ag/Na-glass volume ratio of 4:1 (80 vol.% Ag-20 vol.% glass) and theta ranged between 70° and 90°. The second sample (designated as Sample II(5)), consisted of 35 vol.% Ag - 65 vol.% Na-glass (particle diameters same as Sample II(4)). Theta ranged

between 70° and 90°.

Pre-flight preparation of the TEXUS 2 samples involved certain de-gassing and compaction procedures which the TEXUS 1 samples did not undergo (see Reference (1) for details).

The sample materials were processed under low-gravity conditions in the TEM 01 isothermal furnace. The melt and solidification sequence was similar to the TEXUS 1 sequence. The procedure was slightly altered such that the cartridge was pre-heated to 600 °C prior to TEXUS 2 lift-off (it was heated to 850 °C for TEXUS 1).

Post-flight examination of the low-gravity processed samples led to the following results:

Postflight analysis of samples II(4) and II(5) "...indicate[d] clearly that movement of droplets according to... [Stoke's Law]... [was] not predominant." (2, p. 36) Constituents in sample II(5) (65 vol.% glass) separated and contained a large drop of Ag surrounded by a layer of glass (see Walter, TEXUS 1, sample I(5) for comparison). There were no inclusions within the drop. Sample II(4) (20 vol.% glass) was the same as sample II(5) except there were glass inclusions within the Ag drop. This result "...indicates that particle chains were not sufficiently long to link up each particle with the energetically favorable periphery of the sample (crucible wall). Thus, the preparation of dispersion alloys via spinoidal[sic] decomposition may not be possible, since 3-dimensional network formation is to be expected in case of spontaneous separation of two components having approximately equal volume fraction." (2, p. 36)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Immiscible Fluids, Melt and Solidification,Isothermal Processing, Powder Metallurgy, Metallic Matrix, Glasses, Two-Phase System, Multiphase Media, Model Materials, Binary Systems, Phase Separation, Separation of Components, Stability of Dispersions, Liquid/Liquid Dispersions, Density Difference, Drops, Drop Migration, Thermomigration, Sedimentation, Stokes Sedimentation, Spinodal Decomposition, Buoyancy Effects, Free Surface Elimination, Wetting, Contact Angle, Inclusions, Thermal Soak, Solidification Front Physics, Liquid/Liquid Interface, Solid/Liquid Interface, Bubble Removal, Crucible Effects, Material Interaction with Containment Facility

Number of Samples: two

Sample Materials: Sample II(4)) consisted of Ag (particle diameter of 100 to 200 microns) and Na-glass (particle diameter of 100-160 microns). The sample had an Ag/Na-glass volume ratio of 4:1 (80 vol.% Ag-20 vol.% glass) and theta ranged between 70^{o} and 90^{o}. Sample II(5) consisted of 35 vol.% Ag - 65 vol.% Na-glass (particle diameters same as Sample II(4)). Theta ranged between 70^{o} and 90^{o}.
(Ag*Na*)
Container Materials: molybdenum crucible (TZM alloy) in stainless steel envelope
(Mo*)

Experiment/Material Applications:
See Walter TEXUS 1: Stability of Compound Mixtures/Powder Metallurgy (Composite Materials II: Liquid-Liquid-Gas Systems (this chapter)).

References/Applicable Publications:

(1) Walter, H. U.: Stability of Multicomponent Mixtures Under Microgravity Conditions. In Proceedings of the 3rd European Symposium on Material Sciences in Space, Grenoble, April 24-27, 1979, ESA SP-142, pp. 245-253. (post-flight)

(2) Walter, H. U. and Ziegler, G.: Stability of Multicomponent Mixtures. In Shuttle/Spacelab Utilization Final Report, Project Texus, 1978, Technological Experiments in Micro-gravity, pp. 27-47. (TEXUS 1 and 2) (This paper the same as the Grenoble paper above.)

(3) Walter, H. U. and Ziegler, G.: Rearrangement and Separation Processes During Liquid Phase Sintering Under Microgravity Conditions. In Proceedings of the European Sounding-Rocket, Balloon and Related Research, with Emphasis on Experiments at High Latitudes, Ajaccio, Corsica, April 24-29, 1978, ESA SP-135, pp. 345-352. (post-flight)

(4) Composite Materials II. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 286-287. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 3
Launch Date/Expt Date: April 1980
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2 isothermal furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Stability of Compound Mixtures

This TEXUS 3 experiment was the third in a series of investigations designed by Walter et al. to explore the low-gravity stability of multicomponent liquid-liquid systems during melting, thermal soak, and solidification (see Walter, TEXUS 1, TEXUS 2).

During the Walter's earlier TEXUS 1 study, one of the five samples processed was Ag-Na glass; during his TEXUS 2 study, two of the five were Ag-Na glass. Apparently, during this mission all eight samples processed were Ag-Na glass. The Na glass content of the samples ranged from 2 vol.% to 17 vol.% (see the **Sample Materials** section below).

The samples were processed in an isothermal furnace within the TEXUS Experiment Module TEM 01-2 (previously described under Walter, TEXUS 1). <Note: No other processing parameters (e.g., temperatures) were detailed in the available publications.>

It was reported that during the TEXUS 3 flight, the experiment did not achieve the desired gravity level because of a "...residual spin of the rocket (1 Hz) and centrifugal acceleration of 0.19g resulting therefrom...." (3, p. 9) Despite the undesired accelerations, sample analysis indicated that the lower the amount of minority phase (Na-glass) in the material the greater the stability of the dispersion. The 17% glass sample exhibited complete separation; the 2% glass sample exhibited a very stable dispersion.

Additional results from the TEXUS 3 flight were combined with those from other flight experiments by Walter (TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5). The combined conclusions from these experiments can be found under Walter, TEXUS 5 (this chapter).

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Immiscible Fluids, Melt and Solidification, Isothermal Processing, Thermal Soak, Powder Metallurgy, Multiphase Media, Model Materials, Binary Systems, Metallic Matrix, Glasses, Phase Separation, Separation of Components, Stability of Dispersions, Liquid/Liquid Dispersions, Density Difference, Drops, Drop Migration, Thermomigration, Sedimentation, Buoyancy Effects, Solidification Front Physics, Liquid/Liquid Interface, Solid/Liquid Interface, Rocket Motion, Acceleration Effects, Rocket Despin Failure

Number of Samples: eight
Sample Materials: silver particles/sodium glass particles (2, 4, 6, 8, 11, 13, 15, 17 vol.% Na-glass)
(Ag*Na*)
Container Materials: unknown

Experiment/Material Applications:
See Walter, TEXUS 1.

References/Applicable Publications:

(1) Walter, H. U.: Preparation of Dispersion Alloys- Component Separation During Cooling and Solidification of Dispersions of Immiscible Alloys. In Proc. Workshop on Effect of Gravity on Solidification of Immiscible Alloys, Stockholm, January 18-20, 1984, ESA SP-219, March 1984, p. 47. (post-flight; specific mission(s) unidentified)

(2) Greger, G.: TEXUS and MIKROBA and Their Effectiveness and Experiment Results. Presented at: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP). (identifies rocket failure)

(3) Walter, H. U.: Dispersion Alloys TEXUS-Experiments: TEXUS-V TEM-01 B, TEXUS VII, TEM-01 B. NASA TM-77531, December 1983. (post-flight; in connection with other missions)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 3b
Launch Date/Expt Date: April 1981
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2 isothermal furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Stability of Compound Mixtures (Immiscibles I)

This TEXUS 3b experiment was the fourth in a series of investigations designed by Walter et al. to explore the low-gravity stability of liquid-liquid multi-component systems during melting, thermal soak, and solidification (see Walter, TEXUS 1, TEXUS 2. TEXUS 3). The specific objective of this experiment was to investigate and isolate the various mechanisms which govern component separation in mixtures exhibiting a miscibility gap.

In a terrestrial laboratory, mixtures of Ag and Na-silicate powders were mixed, degassed, and hot compacted creating flight samples of a theoretical density. Reportedly, the glass content was 35 vol.%, 20 vol.%, and 17 vol.%. <Note: The exact number of samples investigated was not reported in the available publications.> The samples were contained in a stainless steel crucible and placed in the TEXUS Experiment Module TEM 01-2 isothermal furnace for processing.

Just prior to the rocket launch, the samples were heated to 750 ^{o}C. After launch, and during the low-gravity period of the flight, the samples were heated to 1150 ^{o}C and soaked at this temperature for 2 minutes. Prior to the end of the low-gravity period and prior to rocket reentry, the samples were cooled to below 850 ^{o}C (Ag melt temperature is 950 ^{o}C).

Post-flight examination of all the flight samples revealed a complete separation of the Ag and Na/silicate glass materials. The glass coated the inside of the crucible; the Ag collected at the core of the samples.

Reportedly, because thermal gradients in the samples were minimized and Ag is an excellent thermal conductor, Marangoni convection should have been negligible. Further, because mutually insoluble components were used, all coarsening mechanisms related to nucleation and growth were avoided. Therefore, it was concluded that separation was observed because of coalescence and wetting due to the high glass volume fraction of the samples. It could be shown, theoretically, that a dispersion containing a minority component of greater than 12% would result in a coherent network of interconnected particles. "This was the case, and the remelting of such dispersions resulted in coalescence and redistribution according to wetting conditions." (3, p. 240)

It was concluded that production of dispersions with immiscible systems is not possible at high volume fractions.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Dispersion Alloys, Immiscible Fluids, Melt and Solidification, Isothermal Processing, Powder Metallurgy, Multiphase Media, Two-Phase System, Model Materials, Binary Systems, Metallic Matrix, Glasses, Phase Separation, Separation of Components, Stability of Dispersions, Liquid/Liquid Dispersions, Density Difference, Drops, Drop Migration, Thermomigration, Sedimentation, Nucleation, Marangoni Convection, Marangoni Convection Diminished, Buoyancy Effects, Wetting, Coarsening, Drop Coalescence, Thermal Soak, Solidification Front Physics, Liquid/Liquid Interface, Solid/Liquid Interface, Crucible Effects, Coated Surfaces, Material Interaction with Containment Facility, Bubble Removal

Number of Samples: unknown
Sample Materials: silver particles/sodium glass particles (Ag*Na*)
Container Materials: stainless steel crucible

Experiment/Material Applications:
See Walter, TEXUS 1 (this chapter).

References/Applicable Publications:

(1) Walter, H. U.: Preparation of Dispersion Alloys-Component Separation During Cooling and Solidification of Dispersions of Immiscible Alloys. In Proc. Workshop on Effect of Gravity on Solidification of Immiscible Alloys, Stockholm, January 18-20, 1984, ESA SP-219, March 1984, p. 47. (post-flight)

(2) Walter, H. U.: Dispersion Alloys TEXUS-Experiments: TEXUS-V TEM-01 B, TEXUS VII, TEM-01 B. NASA TM-77531, December 1983. (post-flight; in connection with other missions)

(3) Immiscibles I. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, p. 240. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 5
Launch Date/Expt Date: April 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1, Chamber B
Builder of Processing Facility: ERNO Raumfahrttechnik, GmbH, Bremen, Germany

Experiment:
Stability of Compound Mixtures (Immiscibles II)

This TEXUS 5 experiment was the fifth in a series of investigations designed by Walter et al. to explore the low-gravity stability of multicomponent liquid-liquid systems during melting, thermal soak, and solidification (see Walter, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b).

Earlier studies by Walter et al. were performed to separate and evaluate gravity-dependent and gravity-independent mechanisms which prevent binary systems with miscibility gaps from producing dispersion alloys when solidified. Results from these previous experiments demonstrated that dispersions require minority component volume fractions below 12%. Therefore, this TEXUS 5 experiment investigated the dispersion stability contributions of these mechanisms using samples which contained less than 12 vol.% of the minority component.

Before the TEXUS flight, six Ag-Na glass powder samples (11, 9, 7, 5, 3, and 1 vol.% glass) and two Al-Bi samples (3.6 and 2.0 vol.% Bi) were de-gassed and compacted prior to incorporation into crucibles. The eight crucibles were then stacked into a single cartridge and configured in the TEM 01-1 furnace (see Walter, TEXUS 1 (this chapter), for a general description of the experimental apparatus).

Just prior to the rocket launch, the cartridge was heated to 750 ^{o}C. During the flight, the TEM 01 furnace produced an isothermal heating range in one half the cartridge and a gradient heating range in the other half. The samples containing 11 vol.%, 9 vol.%, 7 vol.%, and 3 vol.% glass were located in the isothermal

portion of the furnace; the 5 vol.% and 1 vol.% glass and the Al-Bi samples were located within the gradient zone of the furnace (gradients up to 150 $^{o}C/cm$). (Samples were processed directionally in the gradient part of the furnace to investigate the interaction between the moving solidification front and dispersed particles.) Sample processing took place during the low-gravity phase of the rocket flight.

Reportedly, the temperature required for homogenization of the Al-Bi samples was not achieved "...since the samples were located in the cold end of the cartridge and the temperatures were altogether too low." (2, p. 25)

Post-flight analysis of the Ag-Na glass samples indicated that (1) the 11%, 9%, and 7% glass samples experienced partial separation (see Walter, TEXUS 1, TEXUS 2, TEXUS 3, and TEXUS 3b for similar results) and (2) the 5%, 3%, and 1% glass samples had final dispersions which were practically stable. Analysis of these and earlier results led to the following conclusions:

(1) Material transport caused by Marangoni convection could be neglected for the Ag-Na glass system.

(2) The stability of the dispersions increased with the decreasing volume percentage of the minority phase. Stability first appeared when volume percentages of the minority phase were less than 7%. (Stability is defined as the remelted dispersion approximately corresponding to the starting dispersion.)

(3) An impoverished zone formed at the crucible wall because of the formation of particle chains. The width of this impoverished area corresponds to, at a particular concentration, the length of the particle chains.

(4) There was no interaction between the particles and the solidification front.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Dispersion Alloys, Immiscible Fluids, Melt and Solidification, Isothermal Processing, Directional Solidification, Powder Metallurgy, Multiphase Media, Two-Phase System, Model Materials, Binary Systems, Metallic Matrix, Glasses, Bubble Removal, Phase Separation, Separation of Components, Stability of Dispersions, Liquid/Liquid Dispersions, Droplet Dispersion, Particle Dispersion, Density Difference, Drops, Drop Migration, Thermomigration, Mass Transfer, Sedimentation, Marangoni Convection, Marangoni

Convection Diminished, Buoyancy Effects, Wetting, Thermal Gradient, Solidification Front Physics, Liquid/Liquid Interface, Solid/Liquid Interface, Crucible Effects, Material Interaction with Containment Facility, Incomplete Sample Processing

Number of Samples: eight
Sample Materials: six samples: silver particles/sodium-glass particles (glass content from 1 vol.% to 11 vol.%); two samples: Al-3.6 vol.% Bi, Al-2.0 vol.% Bi
(Ag*Na*, Al*Bi*)
Container Materials: molybdenum encased in common steel container
(Mo*)

Experiment/Material Applications:
See Walter, TEXUS 1.

References/Applicable Publications:
(1) Walter, H. U.: Preparation of Dispersion Alloys-Component Separation During Cooling and Solidification of Dispersions of Immiscible Alloys. In Proc. Workshop on Effect of Gravity on Solidification of Immiscible Alloys, Stockholm, January 18-20, 1984, ESA SP-219, March 1984, p. 47. (post-flight)

(2) Walter, H. U.: Dispersion Alloys TEXUS-Experiments (TEXUS-V, TEM-01 B, TEXUS-VII, TEM-01B). NASA TM-77531, December 1983. (post-flight)

(3) Immiscibles II. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 242-243. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 7
Launch Date/Expt Date: May 1983
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2 (isothermal zone furnace)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Stability of Dispersions in Metallic Systems with Miscibility Gaps

This experiment was the sixth in a series of investigations designed by Walter et al. to explore the low-gravity stability of multicomponent liquid-liquid systems during melting, thermal soak, and solidification (see Walter, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5).

"Previous experiments [by Walter et al.] with model systems and powder metallurgically prepared samples had allowed the study of the stability of liquid-liquid dispersions and in particular the influence of the volume fraction. They had shown that the stability of liquid-liquid dispersions can be obtained only for volume fractions below 10%. The aim of this experiment was to check the feasibility of producing metallic dispersions with systems having a miscibility gap in the liquid state." (3, p. 244)

During the TEXUS 7 experiment the following mechanisms were investigated: (a) the effect of the volume fraction of the minor phase, (b) the effect of Marangoni flows, and (c) the mutual effect with the solidification front on the stability of the dispersion.

Prior to the rocket flight, a total of eight samples were stacked in a single cartridge. Samples 1 and 8 were Al-10.0 vol.% In, samples 2 and 7 were Al-7.0 vol.% Bi, samples 3 and 5 were Al-9.0 vol.% Pb, and samples 4 and 6 were Al-5.0 vol.% Pb. Samples 1-4 were configured in the gradient zone of the TEXUS Experiment Module TEM 01-2 furnace and samples 5-8 were configured in the

isothermal zone of the furnace.

Prior to the rocket launch, the samples were heated above their respective miscibility gap critical temperatures and maintained at this temperature for 20 minutes. During the low-gravity phase of the mission, they were cooled and solidified through the miscibility gap. Thermal gradients larger than 1000 ^{o}C/cm were established for all samples in the gradient zone. Reportedly, even the "isothermal" zone samples were subjected to gradients between 10 and 30 ^{o}C/cm. "As an average, cooling through the miscibility gap took 1... [minute] so that the initial dispersion in the liquid state should have been between 10^3 and 10^6 inclusions/cm^3 with diameters up to hundreds of microns." (3, p. 244)

Earlier work had determined that, for a volume percent greater than (approximately) 10% of the minority phase, separation would occur due to seed formation and subsequent coarsening by coalescence (see Walter, TEXUS 5). The metallic dispersions in this TEXUS 7 experiment illustrated that for minority phase volume percentages below 10%, dispersions over the entire sample were obtained although (1) no dispersion with statistical distribution of inclusions was obtained and (2) there was some separation which was primarily due to Marangoni flow (see below).

Post-flight metallographic investigations were conducted on each of the specimens.

Al-10 vol.% In:
Sample 1, which was solidified in the "isothermal zone" and sample 8, which was solidified in the gradient zone, were compared. Reportedly, the two samples had clearly different structures. The gradient sample, which was positioned in the cooling area, solidified "...from below and from the side; the dispersion exhibits correspondingly linear structure...." (2, p. 32) The isothermal sample did not contain similar linear elements, rather "...cloud like circular arrangements of the In-particles... suggest a cellular non-directed solidification...." (2, p. 32) Eutectic structures were found in some areas.

Al-7 vol.% Bi:
As in samples 1 and 8 above, samples 2 ("isothermal") and 7 (gradient) clearly had different structures. Sample 2 contained a cellular structure with an enrichment of Bi at its center. This enrichment was attributed to Marangoni mechanisms. Sample 7 contained linear elements.

Al-9 vol.% Pb:
Comparison of sample 3 ("isothermal") and sample 5 (gradient) again indicated different structures. Sample 3 had "...an unusual, possibility radial symmetrical distribution of lead in

the aluminum matrix... In the center of the sample is a large droplet-shaped lead inclusion... the dispersion of lead becomes distinctly more coarse toward the crucible wall." (2, p. 32) Sample 5, which seems to have solidified quasi-isothermally, had a fairly uniform distribution of lead.

Al-5 vol.% Pb:
Sample 4 ("isothermal") contained a dispersion which became "...increasingly greater toward the top; the particle size... between a few microns and 200 microns." (2, p. 35) Sample 6 (gradient) had a uniform distribution of particles (average diameter near 50 microns).

For all samples (those in the gradient zone as well as those in the "isothermal" zone) a distinct interaction between the inclusions and the solidification front occurred. In the directed solidification samples, inclusions were arranged in chains parallel to the solidification direction, indicating (1) that a cellular rather than planar front was present in the samples and (2) that the inclusions were pushed toward the cell boundaries parallel to the solidification front. In the quasi-isothermal samples, the inclusions also mark the cell boundaries. This was not observed in earlier experiments with the Ag-Na glass system. By estimating the solidification rate for each sample, it was found that "...the droplets interact mutually with the solidification front up to a radius of 50 microns for a solidification rate up to 1/mm/s...." (2, p. 38)

Surface tensions gradients, produced by thermal or solutal gradients in the melt, resulted in Marangoni convection flow around the droplets. This convective flow transported the drops toward a higher temperature region (lower surface energy). By comparing identical samples in the gradient and isothermal sections, Marangoni transport could, reportedly, be qualitatively proven. The samples within the gradient section (gradients of 90 to 130 $^{o}C/cm$) indicated that the hot side (that portion solidified last) was enriched with droplets and pores. In the Al-Bi 7 vol.% and the Al-Pb 9 vol.% samples solidified "isothermally," there was also evidence of Marangoni transport. "Since cooling took place from the outside inward, the highest temperature was in the center of the crucible. Accordingly, transport took place toward the crucible center." (2, p. 40) It was also reported that for sample 1 (solidified isothermally) "...no transport takes place as the result of marangoni convection; either the dispersion is maintained or other transport phenomena led to an energetically stable configuration, in which the better wetting component is deposited at the crucible walls.... Sample 1... shows quite accurately the first named distribution." (2, p. 39) The movement of the minority

phase toward the center of the sample (resulting in a Bi or Pb rich core at the center of the sample) was thus due to convective flows toward the middle. The Al-Pb sample, however, showed large inclusion toward the cell wall. This was probably due to the increased rate of seed growth in this area which resulted from the greater undercooling around the rim of the sample.

It was concluded that the results "...showed that the dispersions needed for technological applications cannot be produced by simply cooling through the miscibility gap even in microgravity. Marangoni transport has to be counteracted or minimized." (3, p. 244)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Dispersion Alloys, Metallic Dispersion, Immiscible Fluids, Melt and Solidification, Isothermal Processing, Directional Solidification, Solidification Rate, Powder Metallurgy, Multiphase Media, Binary Systems, Phase Separation, Separation of Components, Stability of Dispersions, Liquid/Liquid Dispersions, Droplet Dispersion, Particle Dispersion, Density Difference, Drops, Drop Migration, Particle Migration, Particle Transport, Particle Size Distribution, Thermomigration, Mass Transfer, Sedimentation, Coarsening, Drop Coalescence, Particle Coalescence, Surface Tension, Surface Tension Gradients, Surface Energy, Marangoni Convection, Marangoni Movement of Droplets, Marangoni Movement of Droplets, Wetting, Wetting of Container, Crucible Effects, Thermal Gradient, Solutal Gradients, Solidification Front Physics, Planar Solidification Interface, Inclusion and/or Rejection of Particles, Undercooling, Liquid/Liquid Interface, Solid/Liquid Interface, Sample Microstructure, Inclusions, Cellular Morphology, Pores, Eutectics

Number of Samples: eight
Sample Materials: Isothermal samples (given in volume %): (Iso-1) aluminum/indium (90/10%), (Iso-2) aluminum/bismuth (93/7%), (Iso-3) aluminum/lead (91/9%), (Iso-4) aluminum/lead (95/9%); gradient samples (given in volume %): (Grad-5) aluminum/lead (91/9%), (Grad-6) aluminum/lead (95/5%), (Grad-7) aluminum/bismuth (93/7%), (Grad-8) aluminum/indium (90/10%)
(Al*In*, Al*Bi*, Al*Pb*)
Container Materials: All samples contained in alumina

Experiment/Material Applications:
The alloy systems used in this study were selected with regard to the following criteria:
(1) Minority phase maximum vol.% less than 10%.
(2) Miscibility gap temperature range between 600 °C and 1500 °C.
(3) Components vapor pressure low within temperature range of interest.
(4) Large component density deferences.
(5) Low component reactivity.
(6) Problem-free preparation and handling.
(7) Material non-toxicity.
Reportedly, the Al-Pb system has potential as a glide bearing material. Thus, four samples of Al-Pb were processed and only two samples of each of the other systems.

See Walter, Texus 1 (this chapter).

References/Applicable Publications:
(1) Walter, H. U.: Preparation of Dispersion Alloys-Component Separation During Cooling and Solidification of Dispersions of Immiscible Alloys. In Proc. Workshop on Effect of Gravity on Solidification of Immiscible Alloys, Stockholm, January 18-20, 1984, ESA SP-219, March 1984, p. 47. (post-flight)

(2) Walter, H. U.: Dispersion Alloys TEXUS-Experiments: TEXUS-V, TEM-01 B, TEXUS-VII, TEM-01 B. NASA TM-77531, December 1983.

(3) Dispersion Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, ESA SP-1132, pp. 244-245. (post-flight)

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Experiment Origin: Sweden
Mission: TEXUS 2
Launch Date/Expt. Date: November 1978
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Swedish TEXUS experiment module containing the GF 1 directional solidification furnace
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Unidirectional Solidification of a Monotectic Pb-Cu Alloy

This TEXUS 2 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2 (Chapter 14)). The specific objective of the experiment was to study the melting and solidification of an immiscible alloy.

During the TEXUS 2 mission, a Pb-Cu alloy was directionally solidified in one of the gradient furnaces (GF 1) contained in the TEXUS Swedish Module. (See Fredriksson, TEXUS 1, "Dendritic Growth and Segregation Phenomena, Eutectic Al-Cu and Hypereutectic Al-Cu" (Chapter 14), for a detailed description of the furnace.)

Reportedly, post-flight analysis of the thermal data indicated that the sample was heat treated as planned.

No discussion of the sample evaluation could be located in the available publications.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Dispersion Alloys, Metallic Matrix, Monotectic Compositions, Binary Systems, Melt and Solidification, Directional Solidification, Thermal Gradient, Liquid/Liquid Dispersions, Liquid/Liquid Interface, Solid/Liquid Interface, Segregation

Number of Samples: one
Sample Materials: lead-copper alloy
(Pb*Cu*)
Container Materials: unknown

Experiment/Material Applications:
See Fredriksson, TEXUS 2, Segregation Phenomena in Immiscible Alloys, Zn-Bi Alloy (this chapter).

References/Applicable Publications:

(1) Grahn, Civ. Ing. S.: Swedish Experiment Module. In Shuttle/Spacelab Utilization Final Report Project, TEXUS II, 1978, pp. 214-222. (post-flight; discussion of hardware performance).

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Experiment Origin: Sweden
Mission: TEXUS 2
Launch Date/Expt. Date: November 1978
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Swedish TEXUS experiment module containing four ellipsoidal mirror furnaces (MF 1, MF 2, MF 3, MF 9)
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Segregation Phenomena in Immiscible Alloys: Zn-Bi Alloys

This TEXUS 2 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2 (Chapters 14 and 17). The specific objective of this experiment was to study segregation phenomena in immiscible alloys (composite materials).

The experiment was performed in four ellipsoidal mirror furnaces contained in the Swedish TEXUS experimental module. (See Fredriksson, TEXUS 1, Dendritic Growth and Segregation Phenomena, Eutectic Sn-Zn (Chapter 14) for a detailed discussion of the furnace.) During the TEXUS 2 mission, four 6-mm diameter, 5-mm long Zn-Bi alloys (38 wt.% Bi, 24 wt.% Bi, and two 8 wt.% Bi) were melted, heated above the miscibility gap, and isothermally resolidified. One 8 wt.% Bi sample was naturally cooled through the miscibility gap at a rate of 3.6 $^{\circ}$C/sec, and the other was cooled at a lower rate of 2.4 $^{\circ}$C/sec by switching the furnace lamps on and off. The other two samples were naturally cooled at a rate of approximately 3.6 $^{\circ}$C/sec. Corresponding control samples (8 wt.% Bi, 12 wt.% Bi, 16 wt.% Bi, 20 wt.% Bi, 24 wt.% Bi, and 38 wt.% Bi) were melted and resolidified under 1-g conditions. <Note: The exact cooling rates of the 1-g processed samples were not reported.>

Reportedly, all of the samples solidified under 1-g conditions exhibited significant segregation of Bi. Large Bi rich droplets within the Zn matrix could be seen. The formation of these drops was attributed to sedimentation (gravity effects) during cooling.

The two rocket-processed Zn-8 wt.% Bi samples (solidified at different rates) were distinctly different. The 2.4 $^{\circ}$C/sec cooled sample was surrounded by a thin, even layer of Bi and the Zn

matrix contained a homogeneous distribution of particles. (The particles had a mean size of 57.4 microns.) The 3.6 $^{o}C/sec$ cooled sample had a less homogeneous distribution and a somewhat thicker layer of Bi surrounding the Zn matrix. (The mean particle size was also somewhat smaller (49 microns).)

Examination of the low-gravity, 24 wt.% Bi sample revealed a thick layer (approximately 100 microns maximum) of Bi around the outside of the sample. A large Bi-rich area was located near the center of the sample and was in contact with the container at one point. The Bi particles were unevenly distributed and ranged in size from 1 to 500 microns. Some of the larger particles were irregularly shaped indicating particle collision.

The microstructure of the low-gravity, 38 wt.% sample was very similar in terms of maximum particle size to that of the 24 wt.% sample. However, the 38 wt.% sample lacked the large Bi-rich region in the center. The reason for this may have been related to the following: "Some of [the] Bi rich border [had] been pressed out of the crucible, and the Bi-content in the sample [had] thereby been lowered to less than 38%." (1, p. 238) Reportedly, the Bi-rich layers around the samples were attributed to either (1) a sedimentation effect caused by residual gravity levels or (2) an inhomogeneous sample preparation prior to the flight.

The low-gravity results were compared to a theoretical model which considered a diffusion-controlled growth rate within the miscibility gap. It was found that the calculated values of particle size were much lower than the values obtained from the space processed samples, indicating that some sort of collision effect was evident during precipitation.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Dispersion Alloys, Binary Systems, Two-Phase System, Metallic Matrix, Melt and Solidification, Phase Separation, Diffusion, Diffusion-Controlled Growth, Growth Rate, Solidification Rate, Passive Cooling, Sedimentation, Segregation, Separation of Components, Buoyancy Effects, Drops, Droplet Collision, Drop Formation, Precipitation, Homogeneous Dispersion, Liquid/Liquid Dispersion, Particle Dispersion, Particle Size Distribution, Isothermal Processing, Sample Homogeneity, Liquid/Liquid Interface, Solid/Liquid Interface, Sample Microstructure, Crucible Effects, Material Interaction with Containment Facility, Liquid Leakage, Acceleration Effects

Number of Samples: four
Sample Materials: bismuth-zinc. Two samples consisted of 8 wt.% Bi, one consisted of 24 wt.% Bi, and one consisted of 38 wt.% Bi. (Bi*Zn*)
Container Materials: unknown

Experiment/Material Applications:
A major contributing feature to the property of a composite is the size and distribution of the second phase material. It is desirable that this distribution be as homogeneous as possible. When solidifying immiscible alloys under 1-g conditions, separation of the matrix and second phase material occurs (similar to the separation of oil and water) because of sedimentation and buoyancy effects. It had been proposed that solidification of immiscibles under low-gravity conditions should result in a material with a homogeneous distribution of the second phase. However, earlier low-gravity experiments had indicated that this was not the case (e.g., see Reference (4) or Löhberg, SPAR 2, Chapter 17). Rather, a large amount of separation occurred which indicated other factors were present that controlled the separation of immiscible alloys. Some of these factors are masked by gravitational effects and, therefore, cannot be investigated on Earth. It was, therefore, proposed that these effects be investigated and their contributions evaluated under low-gravity conditions.

The specific reasons why the Zn-Bi alloys were selected for this experiment were not detailed in the available publications.

References/Applicable Publications:
(1) Carlberg, T. and Fredriksson, H.: The Influence of Microgravity on the Structure of Bi-Zn Immiscible Alloys. In Proceedings of 3rd European Symposium on Material Sciences in Space, Grenoble, April 24-27, 1979, ESA SP-142, pp. 233-243. (post-flight)

(2) Fredriksson, H.: Solidification Studies. In Shuttle/Spacelab Utilization Final Report, Project TEXUS II, 1978, pp. 146-157. (post-flight)

(3) Carlberg, T., Fredriksson, H., Sunnerkranz, P., Grahn, S., and Stenmark, L.: The Swedish Texus Experiment, A Technical Description and Some Preliminary Results. Proceedings of Esrange Symposium, Ajaccio, April 24-29, 1978, ESA SP-135, June 1978. (preflight TEXUS 2; discusses ellipsoidal furnaces)

(4) Löhberg, K., Dietl, P., and Ahlborn, H.: Segregation and Solidification of Liquid Aluminum-Indium Alloys Under Zero Gravity Conditions. In Space Applications Rocket Program-SPAR II, Final Report, NASA TM-78125, pp. VIII1-VIII44, November 1977. (reference to another experiment only.)

(5) Carlberg, T. and Fredriksson, H.: The Influence of Microgravity on the Solidification of Zn-Bi Immiscible Alloys. In Metallurgical Transactions A, Vol. 11A, October 1980, pp. 1665-1676.

(6) Solidification of Immiscible Alloys (Zn-Bi). In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 250-251. (post-flight)

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Experiment Origin: Sweden
Mission: TEXUS 2
Launch Date/Expt. Date: November 1978
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Swedish TEXUS Experiment Module containing one ellipsoidal mirror furnace (MF 10)
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Isothermal Solidification of Zn-Pb

This TEXUS 2 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2 (Chapters 14 and 17)). The specific objective of the experiment was to study the solidification of an immiscible alloy.

The Zn-Pb alloy was processed in one of ten ellipsoidal mirror furnaces contained in the Swedish Experiment Module of the TEXUS sounding rocket. (See Fredriksson, TEXUS 1, "Dendritic Growth and Segregation Phenomena, Eutectic Sn-Zn" (Cahpter 14), for a detailed description of the furnace.)

Reference (1) indicated that the "...control system response was identical to pre-flight test runs and the performance of the furnace can therefore be regarded as nominal." (1, p. 216).

No discussion of the post-flight sample evaluation could be located in the available publications.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Metallic Matrix, Two-Phase System, Phase Separation, Melt and Solidification, Isothermal Processing, Liquid/Liquid Dispersions, Liquid/Liquid Interface, Solid/Liquid Interface, Segregation

Number of Samples: one
Sample Materials: zinc-lead alloy
(Zn*Pb*)
Container Materials: unknown

Experiment/Material Applications:
See Fredriksson, TEXUS 2, Segregation Phenomena in Immiscible Alloys, Zn-Bi Alloys (this chapter).

References/Applicable Publications:
(1) Grahn, Civ. Ing. S.: Swedish Experiment Module. In Shuttle/Spacelab Utilization Final Report Project, TEXUS II, 1978, pp. 214-222. (post-flight; discussion of hardware performance)

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Experiment Origin: Sweden
Mission: TEXUS 3
Launch Date/Expt. Date: April 1980
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Unknown, probably the ESA/SSC experiment module containing ellipsoidal mirror furnaces
Builder of Processing Facility: (If ESA/SSC module:) Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Immiscible Alloys

This TEXUS 3 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2 (Chapters 14 and 17)). The specific objective of the experiment was to evaluate the processing of immiscible alloys.

The composition of the experiment sample(s) and a description of the processing facility were not detailed in the available publications.

Reportedly, TEXUS 3 did not achieve the desired low-gravity level because of a rocket despin failure. The experiment was reflown on TEXUS 3b (see Fredriksson, TEXUS 3b).

No further information of the TEXUS 3 experiment appears to be available.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Acceleration Effects, Rocket Motion, Rocket Despin Failure

Number of Samples: unknown
Sample Materials: immiscible alloys, specific materials unknown
Container Materials: unknown

Experiment/Material Applications:
unspecified

References/Applicable Publications:
(1) Greger, G.: TEXUS and MIKROBA and Their Effectiveness and Experiment Results. Presented at: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP). (identifies rocket failure)

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Experiment Origin: Sweden
Mission: TEXUS 3b
Launch Date/Expt. Date: April 1981
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: ESA/Swedish Space Corporation experiment module containing ellipsoidal mirror furnaces
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Immiscible Alloys

This Texus 3b experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3 (Chapters 14 and 17)). The specific objective of the experiment was to investigate the precipitation/coalescence process of droplets in a liquid matrix.

Prior to the flight, eleven Zn-Bi samples were prepared for processing. Three different compositions (4 wt.% Bi, 6 wt.% Bi, and 9 wt.% Bi) with varying initial Bi particle distributions were selected. The samples were configured within the isothermal mirror furnaces of the TEXUS Swedish experiment module. (See Fredriksson, TEXUS 1, "Dendritic Growth and Segregation Phenomena, Eutectic Sn-Zn" (Chapter 14), for a detailed description of the furnaces.) <Note: It appears that only ten mirror furnaces were available in the experiment module. It is unclear to the editors if one of the gradient furnaces was used for one of the samples or if an extra mirror furnace was flown on this mission.>

During the mission, the samples were heated to just above the monotectic temperature. They were held at this temperature for a period of time ranging from 10 to 110 seconds. After this hold time, the samples were allowed to cool down passively. <Note: No other details concerning the thermal history of each sample could be located in the available publications.>

It was reported that "The droplet distributions in the flight samples were compared with the droplet distributions in reference samples. It appeared that the coalescence process was much faster than expected when considering only Ostwald ripening and

collision processes. The larger the volume fraction of the droplets, the larger was the difference between predictions and observations. The precipitated liquid even formed a surface layer around some samples in which the collision of the droplets was enhanced accordingly." (1, p. 252)

No other discussion of the results from this experiment could be located at this time.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Dispersion Alloys, Binary Systems, Metallic Matrix, Phase Separation, Melt and Solidification, Drops, Drop Coalescence, Droplet Collision, Droplet Dispersion, Droplet Size, Particle Dispersion, Particle Size Distribution, Liquid/Liquid Dispersion, Precipitation, Ostwald Ripening, Liquid/Liquid Interface, Solid/Liquid Interface, Passive Cooling

Number of Samples: eleven
Sample Materials: Zn-Bi alloys with three different compositions: (1) 4 wt.% Bi, (2) 6 wt.% Bi, and (3) 9 wt.% Bi
(Zn*Bi*)
Container Materials: unknown

Experiment/Material Applications:
See Fredriksson, TEXUS 2, "Segregation Phenomena in Immiscible Alloys, Zn-Bi" (this chapter).

References/Applicable Publications:
(1) The Coalescence Process in Immiscible Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 252-253. (post-flight)

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Experiment Origin: Sweden
Mission: TEXUS 5
Launch Date/Expt. Date: April 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Swedish TEXUS experiment module containing mirror furnaces
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
The Coalescence Process in Immiscible Alloys

This TEXUS 5 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b (Chapters 14 and 17)).

The experiment was one of two solidification studies performed by Fredriksson during the TEXUS 5 mission (see also Fredriksson, TEXUS 5, "Undirectional Solidification of Immiscible Alloys" (this chapter)). The specific objective of this TEXUS 5 experiment was to study the precipitation and coalescence processes of droplets in a liquid matrix.

Prior to the mission, seven Zn-4 wt.% Bi samples were prepared. During this preparation, each sample was quenched at a different rate; thus, each had a different initial Bi particle size. The samples were placed in either graphite or boron-nitride crucibles to allow the study of droplet coalescence dependency on sample/crucible wetting characteristics.

During the low-gravity portion of the mission, the samples isothermally processed by (1) heating above the monotectic temperature, (2) holding at this temperature for a period of time, and (3) cooling passively. <Note: No other discussion of the thermal history was provided.> Reference samples were processed on Earth for comparison.

Post-flight, the droplet distribution in the 1-g and low-gravity samples was compared. It was reported that, as in earlier low-gravity experiments (see e.g., Fredriksson, TEXUS 3b), "...the coalescence process was much faster than expected when considering only Ostwald ripening and collision processes. This was at-

tributed to a Marangoni movement of the droplets. No influence of the crucible material on the coalescence process could be detected. The larger... the initial drop size in the samples, the faster... the coalescence process." (4, p. 254)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Dispersion Alloys, Binary Systems, Metallic Matrix, Melt and Solidification, Drops, Drop Coalescence, Droplet Dispersion, Droplet Size, Particle Dispersion, Particle Size Distribution, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Surface Tension, Marangoni Movement of Droplets, Precipitation, Ostwald Ripening, Segregation, Separation of Components, Isothermal Processing, Passive Cooling, Solid/Liquid Interface, Crucible Effects, Wetting, Wetting of Container, Material Interaction with Containment Facility

Number of Samples: seven
Sample Materials: Zn-4 wt.% Bi
(Zn*Bi*)
Container Materials: Some of the samples were in graphite crucibles, others were in boron nitride crucibles.
(C*, B*N*)

Experiment/Material Applications:
See Fredriksson, TEXUS 2, "Segregation Phenomena in Immiscible Alloys, Zn-Bi" (this chapter).

References/Applicable Publications:

(1) Bergman, A., Fredriksson, H., and Shahani, H.: On the Mechanism of the Coalescence Process in Immiscible Alloys. 26th IAF, International Astronautical Congress, Stockholm, Sweden, October 7-12, 1985, IAF Paper 85-274, 8 pp.

(2) Jönsson, R., Wallin, S., and Holm, P.: The Microgravity Research Program Sweden. AIAA 6th Sounding Rocket Conference, Orlando, Florida, October 26-28, 1982. (post-flight; discusses TEXUS 5 and 7 rocket furnaces)

(3) Bergman, A., Fredriksson, H., and Shahani, H.: The Effect of Gravity and Temperature Gradients on Precipitation in Immiscible Alloys. Journal of Materials Science, 23 (1988), pp. 1573-1579. (post-flight)

(4) The Coalescence Process in Immiscible Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, p. 254. (post-flight)

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Experiment Origin: Sweden
Mission: TEXUS 5
Launch Date/Expt. Date: April 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Swedish TEXUS Experiment Module containing the Gradient Furnace Assembly
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Unidirectional Solidification of Immiscible Alloys

This TEXUS 5 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b (Chapters 14 and 17)).

The experiment was one of two solidification studies performed during the TEXUS 5 mission (see also Fredriksson, TEXUS 5, "The Coalescence Process in Immiscible Alloys" (this chapter)). The specific objective of this TEXUS 5 experiment was to study the effect of a temperature gradient on the precipitation of droplets in an immiscible alloy.

Prior to the flight, two samples were prepared: (1) Cu-36 wt.% Pb and (2) Cu-42 wt.% Pb. After preparation, each sample was placed in its own graphite crucible (0.8 mm wall thickness, 65 mm long, 4 mm inner diameter).

The samples were configured within the furnaces of the Swedish TEXUS Experiment Module such that the bottom of each sample was threaded to a 6 mm copper rod. The copper rod was in contact with a phase-change heat sink. (See Fredriksson, TEXUS 1, "Dendritic Growth and Segregation Phenomena, Eutectic Al-Cu and Hypereutectic Al-Cu" (Chapter 14), for a more detailed description of the furnace facility.)

During the low-gravity portion of the mission (1) both samples were melted, (2) the furnace was switched off, and (3) directional solidification was achieved via heat extraction through the copper rod and phase-change heat sink. Temperatures were measured at three locations in each sample.

Post-flight analysis of the low-gravity, Cu-42 wt.% Pb sample thermal data indicated that a solidification rate of 50 K/cm was achieved at the beginning of the experiment and a solidification rate of 10 K/cm was achieved at the end of the experiment. The interface growth rate was determined to be 0.7 mm/sec. Metallographic analysis indicated the presence of aligned composite structure just above the unmelted portion of the Cu-Pb sample. The amount of aligned structure decreased with distance from the unmelted section. A large lead-rich area which formed at the top of the sample was attributed to segregation effects.

Analysis of the 1-g processed Cu-42 wt.% Pb sample (remelted to 10 mm from the bottom, growth rate approx. = 0.4 mm/sec) revealed a copper-rich structure just above the unmelted section. Above this was a band of lead followed by an aligned structure. Above the aligned structure (34 mm from the bottom of the sample) was a copper dendritic structure. The top portion consisted entirely of a copper-rich dendritic structure.

<Note: The above results were obtained from Reference (3). No other publications which discussed the specific results from the Cu-36 wt.% Pb sample could be located. The remainder of this summary was obtained from Reference (4), which did not distinguish between the two samples.>

"It was observed that, in the space samples, the droplets migrated towards the hotter region during the precipitation process. In the ground processed samples [(melted and solidified in the same furnaces on Earth)], a gravity-induced sedimentation of the droplets occurred.

"A theoretical treatment of the experimental results was performed. The theory relates the movement of the droplets to the temperature dependence of the interfacial tension between the liquid droplets and the liquid matrix." (4, p. 256) (See Reference (3) for a discussion of the theoretical treatment.)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Metallic Matrix, Melt and Solidification, Directional Solidification, Thermal Gradient, Drops, Drop Migration, Thermomigration, Marangoni Movement of Droplets, Precipitation, Interfacial Tension, Liquid/Liquid Interface, Liquid/Liquid Dispersion, Solid/Liquid Interface, Segregation, Sedimentation, Buoyancy Effects, Growth Rate, Solidification Rate, Sample Microstructure, Dendritic Structure

Number of Samples: two
Sample Materials: (1) Cu-36 wt.% Pb and (2) Cu-42 wt.% Pb (Cu*Pb*)
Container Materials: graphite
(C*)

Experiment/Material Applications:
See Fredriksson, TEXUS 2, "Segregation Phenomena in Immiscible Alloys, Zn-Bi Alloys" (this chapter).

The specific reasons why the Cu-Pb alloys were selected for this experiment were not detailed in the available publications.

References/Applicable Publications:
(1) Bergman, A., Fredriksson, H., and Shahani, H.: On the Mechanism of the Coalescence Process in Immiscible Alloys. 26th IAF, International Astronautical Congress, Stockholm, Sweden, October 7-12, 1985, IAF Paper 85-274, 8 pp.

(2) Jönsson, R., Wallin, S., and Holm, P.: The Microgravity Research Program Sweden. AIAA 6th Sounding Rocket Conference, Orlando, Florida, October 26-28, 1982. (post-flight; discusses TEXUS 5 and 7 rocket furnaces)

(3) Bergman, A., Fredriksson, H., and Shahani, H.: The Effect of Gravity and Temperature Gradients on Precipitation in Immiscible Alloys. Journal of Materials Science, 23 (1988), pp. 1573-1579. (post-flight)

(4) Unidirectional Solidification of Immiscible Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 256-257. (post-flight)

(5) Bergman, A., Carlberg, T., Fredriksson, H., and Stjerndahl, J.: The Influence of Gravity on the Solidification of Monotectic and Near Monotectic Cu-Pb alloys. In Materials Processing in the Reduced Gravity Environment of Space, Proceedings of the Materials Research Society Annual Meeting, Boston, Massachusetts, November 1981, pp. 579-592. (preflight, ground-based results)

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Experiment Origin: Sweden
Mission: TEXUS 7
Launch Date/Expt. Date: May 1983
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Swedish TEXUS Experiment Module containing the Gradient Furnace Assembly
Builder of Processing Facility: Unknown, possibly Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Unidirectional Solidification of Immiscible Alloys

This TEXUS 7 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5 (Chapters 14 and 17)). The specific objective of this investigation was to study the effect of a temperature gradient on the precipitation of droplets in an immiscible alloy.

Prior to the TEXUS 7 flight, three Zn-Bi samples with additions of 3% Cu or 3% Mg were prepared. <Note: The specific compositions of the three Zn-Bi samples were not provided in the available publications.> After preparation, the samples were placed in graphite crucibles.

The experimental procedure was the same as that employed during the earlier TEXUS 5 experiment (see Fredriksson, TEXUS 5, "Unidirectional Solidification of Immiscible Alloys" (this chapter)).

It was reported that "During the precipitation process in microgravity, the droplets migrated towards the hotter region of the samples due to the Marangoni effect.

"On Earth, the sedimentation of the droplets occurred as expected but was however balanced by the Marangoni effect when the hot part of the sample was oriented upwards. No difference was observed between the three samples. <Note: Presumably "the three samples" refers to the three TEXUS 7 flight samples.> When compared to previous space experiments, it appeared that the movement of the droplets was faster in the Cu-Pb alloys than in the Zn-Bi alloys. The theoretical treatment of the experimental results [see Reference (3)] relates the motion of the droplets to

their interfacial tension. The different behaviors observed in Cu-Pb and Zn-Bi alloys is due to the different temperature dependence of their interfacial tension." (4, p. 258) <Note: It appears that the "previous space experiments" concerning Cu-Pb alloys refers to a TEXUS 5 experiment by Fredriksson "Unidirectional Solidification of Immiscible Alloys" (this chapter).>

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Ternary Systems, Phase Separation, Melt and Solidification, Directional Solidification, Thermal Gradient, Drops, Precipitation, Drop Migration, Thermomigration, Marangoni Movement of Droplets, Interfacial Tension, Segregation, Sedimentation, Separation of Components, Liquid/Liquid Interface, Liquid/Liquid Dispersion, Solid/Liquid Interface

Number of Samples: three
Sample Materials: zinc-bismuth alloy samples (compositions unknown) with additions of either 3% Cu or 3% Mg
(Zn*Bi*Cu*, Zn*Bi*Mg*)
Container Materials: graphite
(C*)

Experiment/Material Applications:
See Fredriksson, TEXUS 2, "Segregation Phenomena in Immiscible Alloys, Zn-Bi Alloy" (this chapter).

The reasons why Cu and/or Mg were added to the Zn-Bi samples for this experimemt were not discussed in the available publications.

References/Applicable Publications:
(1) Bergman, A., Fredriksson, H., and Shahani, H.: On the Mechanism of the Coalescence Process in Immiscible Alloys. 26th IAF, International Astronautical Congress, Stoçkholm, Sweden, October 7-12, 1985, IAF Paper #85-274, 8pp.

(2) Jönsson, R., Wallin, S., and Holm, P.: The Microgravity Research Program Sweden. AIAA 6th Sounding Rocket Conference, Orlando, Florida, October 26-28, 1982. (post-flight; discusses TEXUS 5 and 7 rocket furnaces)

(3) Bergman, A., Fredriksson, H., and Shahani, H.: The Effect of Gravity and Temperature Gradients on Precipitation in Immiscible Alloys. Journal of Materials Science, 23(1988), pp. 1573-1579. (post-flight)

(4) Unidirectional Solidification of Immiscible Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, p. 258. (post-flight)

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Experiment Origin: Sweden
Mission: TEXUS 12
Launch Date/Expt. Date: May 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Swedish TEXUS experiment module containing a gradient furnace
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Unidirectional Solidification of Zn-Bi Samples

The structure of immiscible alloys after solidification depends upon two effects: (1) the gravity-independent, Marangoni convective movement of the minority phase droplets and (2) gravity-dependent sedimentation or flotation of the droplets. These two phenomena can be distinguished by performing unidirectional solidification experiments under 1-g and low-gravity conditions.

This TEXUS 12 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 10 (Chapters 13, 14 and 17). The specific objective of the experiment was to investigate the two effects mentioned above when the influence of gravity was reduced.

Three Zn-Bi alloys were employed for this study (see **the Sample Materials** section for compositions). Prior to the flight, pure elements (Zn, Bi) were melted in argon using an induction heater. After the liquid alloys were heated above the maximum temperature of the miscibility gap, the center section of the cast ingots were removed and machined to a diameter of 4mm and a length of 60 mm.

The samples were placed in a single graphite tube and configured in the gradient furnace of the Swedish Experiment Module. Surrounding the graphite was a stainless steel tube. Three thermocouples were placed between the stainless steel tube and graphite tube. The bottom of the sample contacted a Cu rod.

During the mission, the samples were melted, the furnace power switched off, and the Cu rod brought into contact with a phase-change heat sink. This procedure resulted in directional solidification of the samples.

Post-flight examination of the samples revealed that "...the crucible[s] were not closed at the very top resulting in boiling of... [zinc] during the [low-gravity] experiment.... The boiling [was] due to the pressure drop in... [the low-gravity environment]." (1, p. 56)

No other publications which discussed the experiment could be located.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Phase Separation, Metallic Matrix, Melt and Solidification, Directional Solidification, Thermal Gradient, Minority Phase, Drops, Flotation of Drops, Drop Migration, Thermomigration, Marangoni Movement of Droplets, Marangoni Convection, Interfacial Tension, Sedimentation, Segregation, Separation of Components, Liquid/Liquid Interface, Liquid/Liquid Dispersion, Solid/Liquid Interface, Sample Microstructure, Boiling, Pressure Drop, Processing Difficulties

Number of Samples: three
Sample Materials: (1) Zn-4 wt.% Bi; (2) Zn-6 wt.% Bi; (3) Zn-8 wt.% Bi
(Zn*Bi*)
Container Material: Graphite contained in stainless steel
(C*)

Experiment/Material Applications:
See Fredriksson, TEXUS 2, "Segregation Phenomena in Immiscible Alloys, Zn-Bi Alloy" (this chapter).

References/Applicable Publications:
(1) Fredriksson, H.: Unidirectional Solidification of Zn-Bi Samples. In TEXUS 11/12 Abschlussbericht 1985, German Publication, p. 56. (post-flight)

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Experiment Origin: Sweden
Mission: TEXUS 14a
Launch Date/Expt. Date: May 1986
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: ESA/Swedish Space Corporation TEXUS experiment module containing the Gradient Furnace Assembly (GFA). The GFA was designed for directional solidification experiments and was originally employed on TEXUS 12.
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Unidirectional Solidification of Zn-Bi Samples

This TEXUS 14a experiment was one in a series of investigations designed by Fredriksson to study low gravity solidification phenomena (see Fredriksson TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 10, TEXUS 12 (Chapters 13, 14 and 17)). The specific objective of the experiment was to investigate the directional solidification of Zn-Bi samples.

Reportedly, an unexpected "wobbling motion" of the TEXUS rocket resulted in uncontrollable vehicle accelerations and the desired low gravity level of 10^{-4} g was not attained. The experiment was reflown on TEXUS 14b (see Fredriksson, TEXUS 14b).

No further information concerning this TEXUS 14a experiment appears to be available.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Metallic Matrix, Binary Systems, Phase Separation, Melt and Solidification, Directional Solidification, Thermal Gradient, Sedimentation, Liquid/Liquid Interface, Liquid/Liquid Dispersion, Solid/Liquid Interface, Rocket Motion, Acceleration Effects

Number of Samples: unknown
Sample Materials: zinc-bismuth, specific compositions unknown (Zn*Bi*)
Container Materials: unknown, possibly boron nitride (B*N*)

Experiment/Material Applications:
unspecified

References/Applicable Publications:

(1) Experimentelle Nutzlast und Experimente TEXUS 14. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 53-55. (in German; post-flight)

(2) Experiment-Module ESA/SSC. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 60-61. (gradient furnace assembly)

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Experiment Origin: Sweden
Mission: MASER 1
Launch Date/Expt. Date: March 1987
Launched From: Esrange, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: One high precision isothermal furnace housed within the Multi-Mission Mirror Furnace Module (M4)
Builder of Processing Facility: SAAB Space, Linköping, Sweden, and The Swedish Space Corporation, Solna, Sweden

Experiment:
Coalescence Process of Immiscible Alloys

This MASER 1 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 10, TEXUS 12, TEXUS 14a (Chapters 13, 14 and 17)). The specific objective of the experiment was to investigate the role of droplet size and Marangoni convection on the coalescence process in an immiscible Zn-Bi system.

Prior to the mission, a single Zn-Bi sample was loaded into a high precision isothermal furnace within the Multi-Mission Mirror Furnace (M4) Module. Three mirror arrays and 30 halogen lamps were configured within the furnace to produce the desired isothermal temperature distribution on the 200-mm long sample. Several thermocouples measured the temperature distribution during processing.

Just prior to rocket launch, the sample was heated to 410 ^{o}C. During the low-gravity phase of the flight, the temperature was increased to 475 ^{o}C and the sample melted under isothermal conditions. It was anticipated that the temperature would then stabilize for 100 seconds. However, a malfunction of the furnace (later attributed to "...a component failure in a standard voltage regulator..." (1, p. 22)) resulted in termination of the melting. Solidification of the sample, via blowing nitrogen gas, proceeded as planned and was completed prior to the re-entry phase of the mission.

Publications which described the post-flight analysis of the sample could not be located.

Key Words: Systems Exhibiting A Miscibility Gap, Immiscible Alloys, Binary Systems, Phase Separation, Melt and Solidification, Isothermal Processing, Drops, Drop Migration, Marangoni Movement of Droplets, Marangoni Convection, Drop Coalescence, Droplet Dispersion, Droplet Size, Particle Size Distribution, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Solid/Liquid Interface, Quench Process, Halogen Lamps, Furnace Malfunction, Incomplete Sample Processing

Number of Samples: one
Sample Materials: ZnBi, composition unknown
(Zn*Bi*)
Container Materials: unknown

Experiment/Material Applications:
See Fredriksson, TEXUS 2, "Segregation Phenomena in Immiscible Alloys: Zn-Bi Alloys" (this chapter).

References/Applicable Publications:

(1) Zaar, J., and Änggard, K.: Maser and Its Effectiveness and Experimental Results. In: In Space '87, Japan Space Utilization Promotion Center (JSUP), October 13-14, 1987. (post-flight; short description)

(2) Jönsson, R.: The Microgravity Program in Sweden - Emphasis on the Materials Rocket Maser. In 15th International Symposium on Space Technology and Science, Tokyo, Japan, May 19-23, 1986, Vol. 2, pp. 2099 - 2110. (preflight)

(3) Zaar, J., Björn, L., and Jönsson, R.: Preliminary MASER 1 Results and the Evolution of the MASER Programme. In Proceedings of the 8th ESA Symposium on European Rocket and Balloon Programmes and Related Research, Sunne, Sweden, May 17-23, 1987, ESA SP-270, pp. 359-361. (post-flight; very short description)

(4) Grunditz, H.: Flight Results of the ESA Experiment Modules in MASER 1. In Proceedings of the 8th ESA Symposium on Rocket and Balloon Programmes and Related Research, Sunne, Sweden, 17-23 May 1987, ESA SP-270, pp. 363- 367. (post-flight)

(5) Grunditz, H.: Experiment Equipment for Metallurgy and Fluid Science Studies Under Microgravity. 37th Congress of the International Astronautical Federation, Innsbruck, Austria, October 4-11, 1986. (preflight)

(6) Jönsson, R.: SSC Microgravity Sounding Rocket Program MASER. 37th Congress of the International Astronautical Federation, Innsbruck, Austria, October 4-11, 1986. (preflight)

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Experiment Origin: Sweden
Mission: TEXUS 14b
Launch Date/Expt. Date: May 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: ESA/Swedish Space Corporation TEXUS experiment module containing the Gradient Furnace Assembly (GFA) (The GFA was designed for directional solidification.)
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Unidirectional Solidification of Zn-Bi Samples

This TEXUS 14b experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 10, TEXUS 12, TEXUS 14a, MASER 1 (Chapters 13, 14 and 17)).

The TEXUS 14b investigation, which was a repeat of Fredriksson's TEXUS 14a experiment, had the same experimental objectives, sample preparation, equipment setup, and processing procedure as did Fredriksson's earlier TEXUS 12 experiment (see Fredriksson, TEXUS 12). Briefly, the objective of the experiment was to investigate how the solidifying structure of immiscible alloys depends on (1) the gravity-independent, Marangoni convective movement of the minority phase droplets and (2) gravity-dependent sedimentation or flotation of the droplets.

It appears that three Zn-Bi alloys were employed for the study (see MATERIALS section for compositions). Prior to the flight, pure elements were melted in argon using an induction heater. After the liquid alloys were heated above the maximum temperature of the miscibility gap, the center section of the cast ingots were removed and machined to a diameter of 4mm and a length of 60 mm.

The samples were placed in graphite tubes, inserted into stainless steel cartridges, and configured in the gradient furnace of the Swedish Experiment Module. Prior to launch, the samples were preheated to 200 ^{o}C. During the low-gravity phase of the mission, (1) the samples were heated to 650 ^{o}C, (2) the furnace power was switched off, and (3) the samples directionally

solidified.

Reportedly, 1-g reference experiments were performed to enable comparison to the low-gravity results.

"The growth rate [of the low-gravity samples] deduced from the temperature recordings could be expressed as follows:

v= 1.28...(* (sqrt z))]mm/sec. <Note: "z" was not defined.> The temperature gradient was evaluated to be around 80 K/cm. The microstructure consisted of Bi-rich droplets precipitated in a monotectic matrix. The number and the average droplet size were evaluated as a function of the distance from the heat sink." (3, p. 260) Reportedly, there were no Bi droplets (TEXUS-processed samples) from the bottom of the samples (nearest to the heat sink) to a distance of 3.5 cm (total sample length of 6 cm). From this point the number and size of the droplets increased with increasing distance up the samples. Examination of the reference samples also revealed no droplets near the bottom. The portion of the reference samples from 2 to 3.5 cm (as measured from the bottom) contained a few large inclusions. Above this region were large numbers of small droplets.

"The difference in droplet distribution between the reference experiments and the flight experiments can be described by the movement of the droplets due to gravity in the reference experiments and the movement of the droplets due to Marangoni convection in the flight experiments. No explanation to the droplet free area at the very bottom of the samples has been found." (1, p. 69)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Monotectic Compositions, Metallic Matrix, Phase Separation, Melt and Solidification, Directional Solidification, Thermal Gradient, Minority Phase, Drops, Flotation of Drops, Drop Migration, Thermomigration, Marangoni Movement of Droplets, Marangoni Convection, Droplet Dispersion, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Precipitation, Sedimentation, Separation of Components, Solid/Liquid Interface, Sample Microstructure, Inclusions

Number of Samples: three
Sample Materials: (1) Zn-4 wt.% Bi, (2) Zn-6 wt.% Bi and (3) Zn-8 wt.% Bi.
(Zn*Bi*)

Container Materials: graphite contained in stainless steel (C*)

Experiment/Material Applications:
See Fredriksson, TEXUS 2, "Segregation Phenomena in Immiscible Alloys, Zn-Bi Alloy" (this chapter).

References/Applicable Publications:

(1) Eliasson, A. and Fredriksson, H.: Unidirectional Solidification of Zn-Bi Samples. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 66-69. (post-flight)

(2) Experiment-Module ESA/SSC. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 60-61. (gradient furnace assembly)

(3) Unidirectional Solidification of Zn-Bi Samples. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 260-261. (post-flight)

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Experiment Origin: Sweden
Mission: MASER 2
Launch Date/Expt. Date: February 1988
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Multi-Mission Mirror Furnace Module (M4): (One of the two available isothermal mirror furnaces was used. The furnace employed three linear-elliptical mirror arrays.)
Builder of Processing Facility: Saab Space, Linköping, Sweden, and the Swedish Space Corporation, Solna, Sweden

Experiment:
A Study of the Coalescence Process of Immiscible Alloys in Large Samples

This MASER 2 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 10, TEXUS 12, TEXUS 14a, MASER 1, TEXUS 14b (Chapters 13, 14 and 17)). The overall objective of the experiment was to study the coalescence process of immiscible alloys. More specifically, the experiment was designed to study the effect of droplet size and Marangoni convection on the coalescence process.

The experiment was performed in the MASER Multi-Mission Mirror Furnace Module (M4). The M4 contained two identical isothermal mirror furnaces, one of which was used for this investigation. (The other furnace was used by Kozma (see Kozma, MASER 2 (Chapter 18)).) The furnace was "...equipped with three linear-elliptical mirror arrays and each array... [was] furnished with a set of ten halogen lamps. These lamps... [were] individually controlled by a microcomputer to give the correct temperature profile on the sample...." (1, p. 13) Three thermocouples in the sample and nine thermocouples in the crucible were positioned to provide a thermal record of the processing. A spring/piston assembly was configured in the sample crucible to (1) insure good thermal contact of the sample and (2) compensate for solidification shrinkage (and, therefore, alleviate material free surfaces).

During the experiment, a single sample of Zn-Bi was processed. The dimensions of this "large" sample were not detailed. It appears that the sample was preheated to 410 ^{o}C just prior to launch (although this is not clearly stated in Reference (3)).

It was reported that once the low-gravity phase of the mission was attained, (1) the molten sample temperature was to be maintained at 425 ^{o}C and (2) quick melting and solidification was to take place at near isothermal conditions. (Solidification was to occur just prior to leaving the low-gravity phase of the mission.)

Post-flight examination of the payload indicated that (1) the M4 operated essentially as expected, (2) controlled heating and cooling of the sample was achieved, and (3) sample material leakage from the crucible into the furnace occurred and zinc condensed on some of the furnace mirrors. Reportedly, the flight sample was 15 mm shorter than expected due to this material leakage.

The flight thermocouple data (from the middle of the sample) indicated that the sample melted approximately 300 seconds after the low-gravity phase had been achieved (or 1150 seconds into the flight). It was noted that this melting time was significantly longer than the time it takes for a ground-based sample to melt (95 seconds). An analysis of the entire thermocouple data available in the sample and crucible indicated that "...the melting starts at one end and passes as a wave along the sample." (3, Appendix 5, p. 8) Cooling of the sample was initiated at 1190 seconds, solidification began at 1210 seconds, and complete solidification was achieved at 1280 seconds (just prior to leaving the low-gravity phase).

It was reported that the flight sample cooled slower than the Earth-processed sample. This slower cooling "...created a... [20 ^{o}C] temperature difference between the sample and the crucible. Large temperature differences also exist[ed] along the sample." (2, Appendix 5, p. 8)

Preliminary metallographic examinations of the sample indicated that an uneven droplet distribution was present. It was reported that this distribution appeared to be "...very influenced by the conditions during the cooling and solidification of the sample. It... [was] also possible that some mixing of the melt... [was] due to the movement involved in the leakage." (3, Appendix 5, p. 9)

Further discussion of the sample analysis was not presented and additional information could not be located which described the overall results of this experiment.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Melt and Solidification, Thermal Gradient, Isothermal Processing, Cooling Rate, Drops, Drop Migration, Marangoni Movement of Droplets, Marangoni Convection, Drop Coalescence, Droplet Dispersion, Droplet Size, Volume Compensation, Free Surface Elimination, Liquid/Liquid Interface, Solid/Liquid Interface, Liquid/Liquid Dispersion, Sample Shrinkage, Contamination Source, Liquid Leakage, Liquid Transfer, Piston System, Halogen Lamps, Processing Difficulties

Number of Samples: one
Sample Materials: Zn-Bi
(Zn*Bi*)
Container Materials: unknown

Experiment/Material Applications:
See Fredriksson, TEXUS 2, "Segregation Phenomena in Immiscible Alloys: Zn-Bi Alloys" (this chapter).

References/Applicable Publications:
(1) Zaar, J. and Dreier, L.: MASER II Final Report. RMLO/1-7, Swedish Space Corporation, August 30, 1988. (post-flight)

(2) Zaar, J. and Änggard, K.: MASER and Its Effectiveness and Experimental Results. In: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP). (short description; preflight)

(3) The Coalescence Process of Immiscible Alloys in Large Isothermal Samples. In MASER II Final Report, RMLO/1-7, Swedish Space Corporation, August 30, 1988, Appendix 5, pp. 8-11. (post-flight)

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Experiment Origin: Sweden
Mission: MASER 2
Launch Date/Expt. Date: February 1988
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Swedish Space Corporation Gradient Furnace (SSC/GF) (This equipment was used during the earlier MASER 1 mission (see Fredriksson, MASER 1).)
Builder of Processing Facility: Swedish Space Corporation, Solna, Sweden

Experiment:
Gradient Solidification of Immiscible Alloys, Zn-Pb

The structure of immiscible alloys is dependent (in part) on (1) convective flow resulting from the imposed thermal gradient and acting gravitational force, (2) Marangoni movement of the droplets as dictated by the thermal gradient, and (3) sedimentation or flotation of the droplets as dictated by the imposed gravitational force.

This MASER 2 experiment was one of a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 10, TEXUS 12, TEXUS 14a, MASER 1, TEXUS 14b (Chapters 13, 14 and 17)). The specific objective of the experiment was to study "...the influence of a thermal gradient on the precipitation of droplets during the solidification of immiscible alloys." (4, p. 262)

Prior to the rocket launch, two 4-mm diameter, 65-mm long Zn-Pb samples were prepared. The first sample consisted of 4 wt.% Pb and the second sample consisted of 2.5 wt.% Pb. Each sample was configured in its own furnace within the MASER Gradient Furnace (GF) Module. (The GF module, which consisted of four furnaces in all, was also used during MASER 2 for another experiment (see Fredriksson, MASER 2, Primary Precipitated Crystal in Directional Solidification Al-Cu (Chapter 14)).) The module was configured such that the sample temperature could be measured at three different locations.

Just prior to the rocket launch, both furnaces were heated to 310 ^{o}C. Approximately 80 seconds after launch, rapid heating of the two samples was initiated. At approximately 130 seconds after

launch, the 2.5 wt.% Pb sample had been heated to 575 ^{o}C and the 4.0 wt.% Pb sample had been heated to 610 ^{o}C (both of these temperatures were above the miscibility gap of the materials). Unidirectional solidification was achieved when a copper plug (which was in contact with the bottom of each sample) was brought into contact with a phase change heat sink after the furnaces were switched off. Heat was extracted through the copper plug and into the heat sink (which contained paraffin wax). (The samples were cooled through the miscibility gap during the low-gravity rocket phase.)

Post-flight plots of the sample temperature-vs-time curves illustrated that the growth rate of the monotectic solidification front followed a parabolic growth law: v = k*sqrt(t) where k = 1.9 (m)(sqrt(sec)) for the low-gravity (10^{-4} g) samples and k =1.6 (m)(sqrt(sec)) for the terrestrial (1-g) reference samples.

Preliminary results as summarized in Reference (3) included the following:

"The temperature gradient was evaluated to be around 70 k/cm for the 10^{-4} g samples and around 80 k/cm for the 1-g samples.

"The microstructure consist[s] of Pb-... [droplets] precipitated in a monotectic matrix." (3, p. 5) <Note: Although it was stated that "There is a clear and observable difference in droplet distribution between the 1g and 10^{-4}g samples" (3, p. 5), no further details of these differences were presented.>

"[The droplet distribution]... can be explained by the movement of the droplets due to gravity in the 1g samples and the movement of the droplets due to Marangoni convection in the 10^{-4}[g] samples." (3, p. 5)

Reference (4) briefly reported the following:

"The metallographic analysis of the samples indicated that the precipitated droplets migrated as expected towards the hottest regions of the samples due to interfacial tension gradients. A theoretical analysis could be performed by correlating the observations with the thermal profile recorded during the flight. The different migration velocities observed between the systems Zn-Pb, Zn-Bi and Cu-Pb [see Fredriksson's work on other MASER and TEXUS flights for information on these other systems] could be related to the different temperature... [dependencies] of the corresponding interfacial tensions. The migration velocity of the droplets increases with an increasing temperature dependence of interfacial tension (accordingly, the higher velocities were observed in the Cu-Pb system).

"In a Zn-Bi sample processed on the ground under thermally stabilizing conditions (hottest region at the top of the sample), the Marangoni effect even balanced the gravity-driven sedimentation of Bi-rich droplets of given size." (4, p. 262)

Very little additional information concerning this experiment could be located at this time.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Metallic Matrix, Binary Systems, Phase Separation, Melt and Solidification, Directional Solidification, Thermal Gradient, Minority Phase, Drops, Droplet Size, Drop Velocity, Particle Distribution, Drop Migration, Thermomigration, Interfacial Tension, Marangoni Movement of Droplets, Marangoni Convection, Flotation of Drops, Droplet Dispersion, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Precipitation, Sedimentation, Buoyancy-Driven Convection, Monotectic Compositions, Sample Microstructure, Solid/Liquid Interface

Number of Samples: two
Sample Materials: (1) Zn-2.5 wt.% Pb, (2) Zn-4 wt.% Pb
(Zn*Pb*)
Container Materials: graphite
(C*)

Experiment/Material Applications:
Direct applications were unspecified for the Zn-Pb system.

References/Applicable Publications:

(1) Zaar, J. and Änggard, K.: Maser and Its Effectiveness and Experimental Results. In: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP), 32 pp. (preflight)

(2) Zaar, J. and Dreier, L.: MASER II Final Report. RMLO/1-7, Swedish Space Corporation, August 30, 1989. (post-flight)

(3) Elisasson[sic], A. and Fredriksson, H.: Gradient Solidification of Immiscible Zn-Pb Alloys in Maser II. In MASER II Final Report, RMLO/1-7, Swedish Space Corporation, August 30, 1989, Appendix 3, 6 pp. (post-flight)

(4) Directional Solidification of Immiscible Alloys Zn-Pb. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 262-263. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 2
Launch Date/Expt. Date: November 1978
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01: Multi-Purpose Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Fundamental Studies of the Manganese-Bismuth System

When a Mn-Bi alloy is in a liquid state, it exhibits a miscibility gap. At 445 °C, an intermetallic, ferromagnetic compound $Mn_{1-x}Bi$ (with X approximately equal to 0.08) will form peritectically. Upon further cooling to 340 °C, the high-temperature $Mn_{1-x}Bi$ phase decomposes and forms the low-temperature, stoichiometric compound MnBi according to the reaction:

$$Mn_{1.08}Bi \longrightarrow MnBi + Mn$$

It has been shown that Mn-Bi alloys of the peritectic composition possess a high magnetic coercive strength. However, sedimentation and buoyancy, combined with the formation of peritectic envelopes around first-created crystals, tend to impede the continuous formation of the alloy structure. As a result, when a manganese-bismuth alloy solidifies under 1-g conditions, manganese accumulates in the upper part of the melt because of density differences between the constituents. This characteristic behavior results in a solidified alloy containing less than 20% of the MnBi phase. On Earth, pure MnBi can only be produced by powder technology, in the form of thin films and small monocrystals.

This TEXUS 2 experiment was the first in a series of investigations designed by Pant et al. to study the effects of the low-gravity environment on the solidification of a manganese-bismuth alloy. The specific objectives of this experiment were to (1) investigate the low-gravity processing of immiscible alloys for

possible technical and commercial applications, (2) determine whether gravity-independent mechanisms (e.g., Marangoni convection) rather than gravity-dependent mechanisms (sedimentation and buoyancy) were responsible for the separation of the constituents of this immiscible alloy, and (3) determine if the Mn-Bi peritectic reaction can proceed freely in the absence of gravity, resulting in either a fine and homogeneous dispersion of MnBi or even a specimen of pure MnBi.

Prior to the rocket flight, two Mn-Bi samples were prepared. Sample 1 was composed of 50 at.% Mn/50 at.% Bi (stoichiometric composition) and sample 2 was composed of 27.34 at.% Mn/72.66 at.% Bi (peritectic composition). The two samples (each 10.7 mm dia. and 19.6 mm long) were placed in Chamber C of the TEXUS Experiment Module TEM 01 Multi-Purpose Furnace. (Chamber C was one of four compartments available in the furnace. The other chambers were not used by this Principal Investigator during this mission.) A thermocouple was located at (1) each end of the cartridge and (2) between the samples.

Prior to launch, the samples were heated to a temperature between 200 and 300 ^{o}C. Throughout the duration of the low-gravity period (approximately 375 seconds at a gravity level less than 10^{-4} g) the samples were melted (1150 ^{o}C) and resolidified. Prior to the rocket re-entry sequence, cooling of the samples to less than 200 ^{o}C was accomplished by a He flow. The cooling rate of sample 1 was 9.5 $^{o}C/s$ while that of sample 2 was 7.5 $^{o}C/s$. The thermocouple readings indicated that each sample was subjected to a significant thermal gradient. A similar ground-based, control experiment was performed for comparison purposes.

Post-flight examination of sample 1 revealed that a significant amount of gaseous inclusions was distributed throughout the sample. <Note: The source of the gas was not detailed.> (No inclusions were present in the corresponding 1-g sample since the gas escaped during melting and solidification.) The gas was unable to escape during the flight experiment and was able to form inclusions within the solidified material. The sample had small areas of segregation around the inclusions (probably due to the interaction between the gas inclusions and melt). However, the rest of the sample showed little gross segregation compared to the 1-g sample. The flight sample also had a 30% increase in the MnBi peritectic phase over the 1-g processed sample (18% by wt. for the 1-g sample versus 23% by wt. for the low-gravity sample). A more uniform distribution of the MnBi phase and smaller MnBi particle size were also evident in the reduced-gravity sample. The particle size results agreed well with theory; in the absence of thermal convection, particle growth is reduced.

Flight sample 2 exhibited results similar to those of sample 1 with the exception that sample 2 had significantly fewer gaseous inclusions resulting in less local segregation.

Coercivity values for the two flight and two ground-based samples were determined at temperatures of 295 K, 77 K, and 4.2 K. Typically, the coercive strength of MnBi magnets is highly dependent on temperature with coercivity values approaching zero at low temperatures. Therefore, magnetization measurements at room temperature were performed using a hystereosograph while those at low temperatures were performed using a He-bath cryostat with a superconducting magnet. The flight samples, in general, had a significant improvement in coercivity values despite the presence of anti-ferromagnetic Mn. This improvement was attributed to (1) the small particle size of the MnBi phase, (2) an increase in the percentage of the MnBi phase, and (3) the homogeneous distribution of the MnBi phase. Other details concerning the coercivity measurement procedures can be found in Reference (1), p. 58.

Reportedly, this study did not determine if continuous peritectic structure formation can be achieved in the low-gravity environment. However, ground-based experiments, using a new method, where "...a quasi infinitely thin molten zone migrates through a specimen consisting of a Mn-Bi alloy with 20% Mn" (1, p. 59) indicated that it is possible (within a limited area of the sample) to produce pure MnBi. Reportedly, with more time available under reduced-gravity conditions, it may be possible to produce a pure MnBi intermetallic.

The following conclusions were reported:

(1) Gravity-independent forces had no effect on the solidification of the samples.

(2) The low-gravity samples had a significant increase in the amount of ferromagnetic MnBi phase.

(3) The low-gravity samples had a smaller particle size and more uniform distribution of the MnBi phase than the 1-g processed samples.

(4) Magnetic property improvement of the low-gravity samples was due to improved structure.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Metallic Matrix, Intermetallics, Ferromagnetic, Peritectic Reaction, Stoichiometric Compound, Magnetic Composites, Magnetic Properties, Coercive Strength, Phase Separation, Melt and Solidification, Thermal Gradient, Density Difference, Sedimentation, Buoyancy Effects, Segregation, Separation of Components, Dispersion, Homogeneous Dispersion, Particle Dispersion, Particle Distribution, Particle Size Distribution, Particle Growth, Inclusions, Marangoni Convection, Marangoni Movement of Droplets, Liquid/Vapor Interface, Liquid/Liquid Interface, Liquid/Liquid Dispersion, Solid/Liquid Interface, Buoyancy Effects Diminished, Sample Microstructure, Gas Formation, Bubble Formation, Quench Process, Cooling Rate

Number of Samples: two
Sample Materials: Manganese-bismuth alloys: Sample 1: 50 at.% Mn/50 at.% Bi (stoichiometric composition); Sample 2: 27.34 at.% Mn/72.66 at.% Bi (peritectic composition)
(Mn*Bi*)
Container Materials: molybdenum alloy TZM
(Mo*)

Experiment/Material Applications:
It has been shown that Mn-Bi alloys of the peritectic composition possess a high magnetic coercive strength. This property is due to the presence of the intermetallic MnBi phase. However, when melted on Earth, the Mn tends to rise to the top of the crucible. This separation results in (1) a decrease in the amount of Mn available for the formation of the MnBi intermetallic and (2) a decrease in the coercive strength since Mn is anti-ferromagnetic. The production of large monocrystals of MnBi would be of major importance for further investigations in the field of magnetics and magneto-optics.

The compositions of the two Mn-Bi samples processed under low-gravity conditions were selected as "...an appropriate supplement to the Mn-Bi experiment with eutectic composition (2.2 at.% Mn; 97.8 at.% Bi) which was carried out during the ASTP mission." (1, p. 50) (See Larson, ASTP (this chapter) to review the results of the ASTP sample.)

References/Applicable Publications:

(1) Pant, P.: Fundamental Studies in the Manganese-Bismuth System. Shuttle/Spacelab Utilization Final Report Project TEXUS II, 1978, pp. 48-61. (post-flight)

(2) Pant, P., Krupp, F., Wijingaard, J., and Haas, C.: Physical Properties of MnBi Specimens Produced in Microgravity. 27th Aerospace Sciences Meeting, January 9-12, 1989, Reno, Nevada. (post-flight)

(3) Pant, P.: Grundlagenuntersuchungen im System Mangan-Wismut unter verminderter Schwerkraft im Rahmen des TEXUS-II-Projektes. Tech. Mitt. Krupp Forsch. Ber. Band 37 (1979), H. 2, pp. 70-78.

(4) Pant, P.: Fundamental Studies on the Manganese-Bismuth System in Microgravity. Proc. 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, pp. 335-338. (post-flight)

(5) Pant, P.: Poster presentation of the results of the microgravity experiments in TEXUS II, STS 007, and STS 025. Conference on Gravitational Effects on Material Processes, August 17-21, 1987, New London, Hampshire. (post-flight)

(6) Pant, P., Wijingaard, J. H., and Haas, C.: Physical Properties of MnBi-Specimens Produced in Microgravity. <Note: The publication status of this document is unclear at this time. Reportedly, the document was to be published in Journal of Spacecraft and Rockets.> (post-flight)

(7) Input received from Principal Investigator P. Pant, June 1989.

(8) Fundamental Studies in the Manganese-Bismuth System. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 248-249. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: West German Get Away Special (GAS) MAUS Canister DG-206A; SPAS STS Deployed Satellite
Volume of Canister: 5.0 cubic feet
Location of Canister: The West German Shuttle Pallet Satellite (SPAS-01)
(SPAS was a small experiment carrier initially configured in the STS payload bay but later deployed into orbit by the Canadian Remote Manipulator Arm. The carrier was retrieved prior to the end of the shuttle mission.)
Primary Developer/Sponsor of DG-206A: Deutsche Forschungs-und Versuchsanstalt für Luft-und Raumfahrt (DFVLR), Germany/Messerschmitt-Boelkow-Blohm (MBB-ERNO), Bremen, Germany <Note: The DFVLR is now called the Deutsche Forschungsanstalt für Luft- und Raumfahrt (DLR).>
Processing Facility: TEXUS Experiment Module TEM 01 (isothermal four-chamber furnace)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Fundamental Studies in the Manganese-Bismuth System (DG-206)

This STS-007 experiment was the second in a series of investigations designed by Pant et al. to study the effects of the low-gravity environment on the solidification of a Mn-Bi alloy (see Pant, TEXUS 2). The experiment was flown as part of the German MAUS payload onboard the space shuttle (MAUS payload DG-206).

Earlier work by Pant during the TEXUS program indicated that processing Mn-Bi alloys under reduced-gravity conditions resulted in (1) a significant increase in the formation of MnBi phase, (2) a smaller MnBi particle size and more uniform distribution of the MnBi phase, and (3) magnetic property improvement over 1-g processed samples. It was believed that processing times longer than that available during a sounding rocket flight would result in samples with a higher MnBi phase content (and thus improved magnetic properties).

During the mission, eight Mn-50 at.% Bi samples were to be processed in four chambers of the TEM 01 isothermal furnace. The specimens were to be heated to 1150 °C, cooled down to the peritectic temperature (455 °C), and held at this temperature for up to 3 hours. However, because of an "...electromagnetic fault, it was only possible to melt the two specimens in chamber A and cool them down... [uncontrollably]." (3, p. 336)

Post-flight examination of the two melted specimens revealed the presence of very large Mn particles. The formation of these particles was attributed to the slow cooling rate. Extremely large MnBi crystals were also present in the samples.

Reportedly, the design of the MAUS equipment apparatus did not allow the use of high cooling rates. High cooling rates are necessary for the production of very fine Mn particles, and, in turn, fine Mn particles are necessary for the peritectic reaction (the formation of MnBi phase) to freely proceed. Therefore, it was proposed that during the next flight experiment the following time-temperature profile be utilized:

"Heating to the peritectic temperature with subsequent temperature oscillation around the peritectic temperature (delta T = 20°C) over a period of 180 min. and 60 min." (3, p. 336)

No further information concerning this experiment could be located at this time.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Metallic Matrix, Intermetallics, Peritectic Reaction, Stoichiometric Compound, Magnetic Composites, Magnetic Properties, Coercive Strength, Melt and Solidification, Isothermal Processing, Cooling Rate, Phase Separation, Density Difference, Sedimentation, Buoyancy Effects, Segregation, Separation of Components, Dispersion, Homogeneous Dispersion, Particle Dispersion, Particle Distribution, Particle Size Distribution, Liquid/Liquid Interface, Liquid/Liquid Dispersion, Solid/Liquid Interface, Sample Microstructure, Hardware Malfunction, Processing Difficulties, Sample Not Processed As Planned

Number of Samples: eight
Sample Materials: manganese-bismuth (50 at % Mn and 50 at % Bi) (Mn*Bi*)
Container Materials: unknown

Experiment/Material Applications:
See Pant, TEXUS 2.

References/Applicable Publications:

(1) STS-7 Cargo Systems Manual: SPAS-01, JSC-18350 Final Version, NASA JSC, December 21, 1982. (preflight)

(2) Pant, P., Krupp, F., Wijingaard, J., and Haas, C.: Physical Properties of MnBi Specimens Produced in Microgravity. 27th Aerospace Sciences Meeting, January 9-12, 1989, Reno, Nevada, AIAA 89-030. (post-flight)

(3) Pant, P.: Fundamental Studies on the Manganese-Bismuth System in Microgravity. In Proc. 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986. (post-flight)

(4) Pant, P.: Poster presentation of the results of the microgravity experiments in TEXUS II, STS 007, and STS 025. Conference on Gravitational Effects on Material Processes, August 17-21, 1987, New London, Hampshire. (post-flight)

(5) Baum, D., Otto, G., and Vits, P.: MAUS-A Flight Opportunity for Automated Experiments Under Microgravity Conditions. Acta Astronautica, Vol. 11, No. 3-4, pp. 239-245, 1984. (no results reported)

(6) Baum, D., Stolze, H., and Vits, P.: First Flight Data From MAUS Payloads on STS 7 and STS 11. 35th Congress of the International Astronautical Federation, October 7-13, 1984, Lausanne, Switzerland, IAF-84-137, 11 pp. (post-flight)

(7) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report # EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special Canister mission history)

(8) Input received from Principal Investigator P. Pant, June 1989.

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #18, STS-025 (STS 51-G, Discovery)
Launch Date/Expt. Date: June 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: West German Get Away Special (GAS) MAUS Canister DG-206B (Also designated as NASA Get Away Special (GAS) Canister G-028)
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-028/DG-206B: Deutsche Forschungs- und Versuchsanstalt für Luft-und Raumfahrt (DFVLR), Germany/Messerschmitt-Boelkow-Blohm (MBB-ERNO), Bremen, Germany <Note: The DFVLR is now called the Deutsche Forschungsanstalt für Luft- und Raumfahrt (DLR).>
Processing Facility: TEXUS Experiment Module TEM 01 (isothermal four-chamber furnace)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Fundamental Studies of the Manganese-Bismuth System

This STS-025 experiment was the third in a series of investigations designed by Pant et al. to study the effects of the low-gravity environment on the solidification of a Mn-Bi alloy (see Pant, TEXUS 2, STS-007). The experiment was flown as part of the German MAUS program onboard the U.S. space shuttle.

The specific objectives of the investigation were to (1) determine whether other mechanisms, besides those due to gravity effects, were responsible for the separation of phases in the Mn-Bi alloy and (2) determine if the Mn-Bi peritectic reaction can proceed freely in the absence of gravity (see Pant, TEXUS 2).

Typically, homogenization of MnBi specimens ,followed by rapid cooling at the peritectic temperature results in a fine-grained structure of Mn and MnBi within a Bi matrix. The available MAUS hardware, however, was not equipped with a cooling gas supply and rapid cooling was not possible. Therefore, a different time-temperature profile was used (see Pant, STS-007, for a discussion of this procedure).

During the mission, six Mn-Bi samples (50 at.% Mn and 50 at.% Bi) were melted and solidified in three chambers of the TEM 01 isothermal furnace. Before solidification, the samples were "...subjected to temperature oscillations between 450 ^{o}C and 470 ^{o}C with 10 minute intervals for three hours." (9, p. 5) (The peritectic temperature for the Mn-Bi system is 455 ^{o}C.)

Post-flight examination of the processed samples confirmed the results from an earlier TEXUS 2 experiment by Pant et al. Gravity independent Marangoni convection (resulting from differences in interfacial energies between the MnBi particles) did not produce segregation in the samples. Despite the lack of rapid cooling, and thus the absence of the homogenization process, the flight samples contained an extensive amount of MnBi formation. Low temperature magnetization measurements (see Reference (9) for experimental details) revealed that the samples consisted of up to 44.4 mass percent MnBi.

The large amount of MnBi phase present in the procured materials permitted the samples to undergo subsequent thermomechanical treatment of the materials. Extrusion at 220 ^{o}C, produced specimens which were 2 mm in diameter and up to 80 mm in length. A major result of this treatment was very surprising: the extruded flight material consisted of nearly 100% MnBi phase. It was reported that this result was due to "...the high pressures achieved during extrusion just below the melting point (a temperature where Bi atoms already have high mobility) [which] induces rapid diffusion and subsequent formation of the MnBi phase." (9, p. 6) (Mn-Bi alloys solidified on Earth cannot typically by subjected to this type of thermomechanical process; differing hardnesses of the three phases (Mn, Bi, MnBi) and an abundance of Mn in the system make the terrestrial samples difficult to deform.)

Low temperature magnetization measurements revealed that the extruded flight samples contained up to 95.7% MnBi phase. Subsequent X-ray diffraction measurements and cell parameter determinations supported the presence of large amounts of the MnBi phase. Room temperature magnetic measurements demonstrated that extrusion results in the formation of the MnBi phase and "...also imparts a texture which yields an improvement in the magnetic values in a preferred direction." (9, p. 8)

Electrical resistivity measurements (at temperatures between 4.2 K and 300 K) were made of the flight and extruded flight specimens and were compared to the TEXUS 2 flight specimens. Because the residual resistivity at T = 0 is due to atomic disorder and foreign atoms within the crystalline phases, this measurement provides an indication of the quality of the specimen. Reportedly, the samples processed on the shuttle had lower

residual resistivities than the TEXUS 2 specimens. The extruded flight sample had the lowest residual resistivity of the three. The residual resistivity of the low-gravity processed material was compared to that from single crystals of MnBi (see Reference (9) for source of the MnBi single crystal residual resistivity data). It was determined that values for the extruded flight material were one to two orders of magnitude higher than those for single crystals of MnBi. This was attributed to a large amount of atomic disorder and defects within the extruded flight specimen caused by the extrusion process.

The low temperature resistivity of the extruded, 95% MnBi sample illustrated a T^2 dependency which could indicate a number of scattering processes: s-d electron-electron scattering, magnon scattering, and impurity (interstitial) scattering. However, the residual resistivity measurement and the large value of the T^2 multiplier indicated that magnon scattering is the process in effect. (Reference (6) also includes discussions on the Seebeck and Hall effects.)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Metallic Matrix, Intermetallics, Peritectic Reaction, Stoichiometric Compound, Magnetic Composites, Magnetic Properties, Melt and Solidification, Phase Separation, Density Difference, Sedimentation, Buoyancy Effects, Segregation, Separation of Components, Dispersion, Homogeneous Dispersion, Particle Dispersion, Particle Distribution, Interfacial Energy, Marangoni Convection, Marangoni Convection Diminished, Diffusion, Thermal Oscillations, Cooling Rate, Liquid/Vapor Interface, Liquid/Liquid Interface, Solid/Liquid Interface, Sample Microstructure, Hardness

Number of Samples: six
Sample Materials: 50 at.% Mn and 50 at.% Bi
(Mn*Bi*)
Container Materials: molybdenum alloy TZM
(Mo*)

Experiment/Material Applications:
See Pant, TEXUS 2.

References/Applicable Publications:

(1) Cargo Systems Manual: GAS Annex for STS 51-G, JSC-17645 51-G, Rev.-A, March 20, 1985. (short description; preflight)

(2) Otto, G. H. and Baum, D.: Material Sciences Experiments Under Microgravity Conditions with MAUS. In Goddard Space Flight Center's 1985 Get Away Special Experimenter's Symposium, October 8-9, 1985, NASA CP-2401, pp. 101-108. (preflight)

(3) STS 51-G Press Kit, NASA Press Release 85-83, June 1985. (preflight)

(4) Kolcum, E. H.: Fuel Contaminant Threatens Delay in Shuttle Launch, AW&ST, June 17, 1985. (preflight)

(5) Otto, G. H. and Staniek, S.: Recent Results from MAUS Payloads. In Goddard Space Flight Center's 1986 Get Away Special Experimenter's Symposium, October 7-8, 1986, pp. 207-213, NASA CP-2438. (post-flight)

(6) Pant, P., Krupp, F., Wijingaard, J., and Haas, C.: Physical Properties of MnBi Specimens Produced in Microgravity. 27th Aerospace Sciences Meeting, January 9-12, 1989, Reno, Nevada, AIAA 89-0303. (post-flight)

(7) Pant, P.: Fundamental Studies on the Manganese-Bismuth System in Microgravity. Proc. 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, pp. 335-338. (post-flight)

(8) Pant, P.: Poster presentation of the results of the microgravity experiments in TEXUS II, STS 007, and STS 025. Conference on Gravitational Effects on Material Processes, August 17-21, 1987. (post-flight)

(9) Pant, P., Wijngaard, J. H., and Haas, C.: Physical Properties of Mn-Bi Specimens Produced in Microgravity. <Note: The publication status of this document is unclear at this time. Reportedly, the document was to be published in Journal of Spacecraft and Rockets.> (post-flight)

(10) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, The NASA GAS Team, 1989, p. 29. (post-flight; very brief description)

(11) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report # EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special Cansiter mission history)

(12) Input received from Principal Investigator P. Pant, June 1989.

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Current Work Address Unknown

Principal Investigator(s): Pirich, R. G. (1), Larson, D. J., Jr. (2)
Co-Investigator(s): Unknown
Affiliation(s): (1,2) Grumman Aerospace Corporation, Bethpage, New York

Experiment Origin: USA
Mission: SPAR 6
Launch Date/Expt. Date: October 1979
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Automated Directional Solidification System (ADSS) (The ADSS was designed to insure that furnace translations resulted in a total ADSS momentum of zero.) <Note: The ADSS was later called the Automated Directional Solidification Furnace (ADSF).>
Builder of Processing Facility: General Electric, Pennsylvania. <Note: It is not clear if this General Electric division was located in Philadelphia, Pennsylvania or King of Prussia, Pennsylvania.>

Experiment:
Directional Solidification of Magnetic Composites (76-22)

Directional solidification of eutectic Bi/MnBi results in an ensemble of MnBi rods, dispersed in a Bi terminal matrix solution. The eutectic is sensitive to thermosolutal convections within the melt. These convections result in growth rate fluctuations and subsequently, microstructural variations. These variations often lead to changes in rod diameter, interrod spacing, electronic/magnetic properties, etc.

This SPAR 6 experiment was the first in a series of investigations designed by Pirich and/or Bethin et al. to study the low-gravity directional solidification of a Bi/MnBi eutectic. It was suspected that a reduction of thermosolutal convection would be realized during the experiment, thus allowing an assessment of the role of gravity driving microstructural variations.

Ninety minutes prior to the rocket launch, the four furnaces within the Automated Directional Solidification System (ADSS) were preheated. (Each furnace contained a single Bi/MnBi sample.) Approximately 120 seconds after launch, the low-gravity phase was attained, and commencement of solidification took place. <Note: The specific preheating and processing temperatures of each sample were not clearly stated.> The four samples were solidified in a Bridgman-Stockbarger configuration; the thermal gradients maintained at about 100 ^{o}C/cm, the furnace speeds regulated at about 30 cm/hr.

Reportedly, one of the ampoules broke at launch. Analysis of the three other flight samples indicated that very uniform, cooperative growth had occurred during the low-gravity processing. Comparison of flight samples with similarly processed ground-based samples indicated that the flight samples exhibited significant reductions in (1) mean rod diameter, (2) interrod spacing and (3) bulk volume fractions. Thermal profiles and magnetic properties of ground and flight samples were very similar.

Many other details concerning the sample analyses are discussed in Reference (1).

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Eutectics, Magnetic Composites, Magnetic Properties, Ferromagnetic, Electrical Properties, Binary Systems, Melt and Solidification, Directional Solidification, Bridgman Technique, Thermal Gradient, Growth Rate, Furnace Translation, Solutal Gradients, Thermosolutal Convection, Buoyancy-Driven Convection, Dispersion, Particle Dispersion, Liquid/Liquid Interface, Solid/Liquid Interface, Planar Solidification Interface, Interrod Spacing, Sample Microstructure, Rod Structure, Rocket Vibration, Acceleration Effects, Payload Survivability

Number of Samples: four
Sample Materials: bismuth/bismuth-manganese samples: 0.72 +/- 0.03 wt.% Mn resulting in a MnBi volume fraction of 3.18 +/- 0.09.
(Bi*/Mn*Bi*)
Container Materials: quartz
(Si*O*)

Experiment/Material Applications:
"The Bi/MnBi eutectic was chosen because its microstructure is characterized by a regular rod eutectic morphology when grown by plane-front solidification with cooperative growth...." (1, p.VI-1) Further, the system is "...sensitive to thermal and solutal instabilities produced by convective flows. In addition, the equilibrium phase of MnBi is highly ferromagnetic and its magnetic properties can be used to characterize the effect of solidification processing and convection on rod size, shape, and alignment." (1, p. VI-1)

References/Applicable Publications:

(1) Pirich, R. G. and Larson, D. J.: SPAR VI Technical Report for Experiment 76-22 - Directional Solidification of Magnetic Composites. In Space Processing Applications Rocket Project SPAR VI Final Report, NASA TM-82433, pp. VI-i - VI-58. (post-flight)

(2) Pirich, R. G., Larson, D. J. Jr., and Busch, G.: SPAR and ASTP Studies of Plane Front Solidification and Magnetic Properties of Bi/MnBi. AIAA 18th Aerospace Sciences Meeting, January 14-16, 1980, Pasadena, California, AIAA-80-0119, 6 pp. (post-flight)

(3) Pirich, R. G., Larson, D. J., and Busch, G.: Studies of Plane-Front Solidification and Magnetic Properties of Bi/MnBi. AIAA 80-0119R, AIAA Journal, Vol. 19, No. 5, May 1981. (post-flight)

(4) General Electric Company, Space Sciences Laboratory, Operating Manual for Automated Directional Solidification System. Prepared for NASA under Contract NAS8-35136, June 1978. (processing facility)

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Principal Investigator(s): Pirich, R. G. (1)
Co-Investigator(s): Unknown
Affiliation(s): (1) Grumman Aerospace Corporation, Bethpage, New York

Experiment Origin: USA
Mission: SPAR 9
Launch Date/Expt. Date: January 1981
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Automated Directional Solidification System (ADSS)/Automated Directional Solidification Furnace (ADSF-1) (The ADSS was designed to insure that furnace translations resulted in a total ADSS momentum of zero.)
Builder of Processing Facility: Original configuration built by General Electric, King of Prussia, Pennsylvania

Experiment:
Directional Solidification of Magnetic Composites (76-22/2)

This SPAR 9 experiment was the second in a series of investigations designed by Pirich and/or Bethin et al. to study the low-gravity directional solidification of a Bi/MnBi eutectic (see Pirich, SPAR 6).

During the mission, four samples were solidified in a Bridgman Stockbarger configuration in the Automated Directional Solidification System (ADSS). A planar solidification interface was produced at approximately 265 ^{o}C; a furnace gradient of 100 ^{o}C/cm was maintained. While a furnace velocity of 30 cm/h was employed on SPAR 6, a 50 cm/h rate was employed during this SPAR 9 mission.

Flight samples were compared to similarly produced ground-processed samples. Reportedly, the morphology of the flight samples was striking. "As was observed during the SPAR VI experiment conducted at a lower solidification velocity of 30 cm/h, the MnBi rod diameter and interrod spacing distributions were significantly smaller, approximately 50%, for the low gravity samples. Accompanying the smaller MnBi rod diameters, the smallest ever achieved in the Mn-Bi system, was an increase in permanent magnet properties. For example, the intrinsic coercivity reached greater than 97% of the theoretical maximum, the largest ever observed in the Mn-Bi system. Also, in-situ thermal measurements during solidification showed a statistically significant lower solidification temperature in low gravity compared with one gravity with an increased interfacial undercooling of about 5.5 ^{o}C. In addition, a lower volume fraction of dispersed MnBi, on the order of 8% was indicated for most of the low

gravity interval of solidification. This suggests a change in the equilibrium diagram in the vicinity of the eutectic composition which is in qualitative agreement with the increased undercooling noted during low gravity solidification. Gravitationally induced convection is suggested to explain the morphological differences between one and low gravity solidification." (1, p. III-iii)

Many other details concerning sample analyses can be found in Reference (1).

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Metallic Matrix, Eutectics, Magnetic Composites, Magnetic Properties, Ferromagnetic, Electrical Properties, Binary Systems, Melt and Solidification, Directional Solidification, Bridgman Technique, Thermal Gradient, Undercooling, Solutal Gradients, Thermosolutal Convection, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Dispersion, Particle Dispersion, Solidification Rate, Liquid/Liquid Interface, Solid/Liquid Interface, Planar Solidification Interface, Growth Rate, Furnace Translation, Interrod Spacing, Sample Microstructure, Rod Structure

Number of Samples: four
Materials: bismuth/bismuth-manganese samples: 0.72 +/- 0.03 wt.% Mn resulting in a MnBi volume fraction of 3.18 +/- 0.09.
(Bi*/Mn*Bi*)
Container Materials: quartz
(Si*O*)

Experiment/Material Applications:
See Pirich, SPAR 6

References/Applicable Publications:
(1) Pirich, R. G.: SPAR IX Technical Report for Experiment 76-22 Directional Solidification of Magnetic Composites. In Space Processing Applications Rocket (SPAR) Project, SPAR IX Final Report, NASA TM-82549, pp. III-i - III-46, January 1984. (post-flight)

(2) DeCarlo, J. L. and Pirich, R. G.: Directional Solidification of Bi-Mn Alloys Using an Applied Magnetic Field. Final Report, 1 January 1, 1984-December 31, 1986 (Grumman Research Corporation), NASA CR-179127, 46 pp. (related ground-based research)

(3) General Electric Company, Space Sciences Laboratory, Operating Manual for Automated Directional Solidification System. Prepared for NASA under Contract NAS8-31535, 1978. (processing facility)

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Experiment Origin: France
Mission: SPAR 9
Launch Date/Expt. Date: January 1981
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: General Purpose Rocket Furnace (Two of the three available heated cavities in the GPRF were dedicated to this experiment.)
Builder of Processing Facility: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Directional Solidification of Immiscible Aluminum Indium Alloys (Experiments 76-51/1 and 76-51/2)

This SPAR 9 experiment was the first in a series of investigations designed by Potard et al. to study low-gravity directional solidification. The specific objectives of the investigation were to (1) analyze the mechanisms which control the solidification process of an immiscible alloy and (2) obtain a regularly dispersed structure of a hypermonotectic composition.

Two of the three cavities of the SPAR 9 General Purpose Rocket Furnace (GPRF) were dedicated to this experiment. Each cavity contained two silicon carbide cartridges. In the first cavity, each cartridge contained a hypermonotectic composition Al-32.08 wt.% In sample. In the second cavity, one cartridge contained a sample of the hypermonotectic composition and the other cartridge contained a monotectic composition Al-16.0 wt.% In sample. It was noted that "Because of the non-regular shape of the crucible..., total [ground-based] filling was not possible. Consequently, large free volumes were unavoidable. This drawback may lead to perturbations of the thermal field and of liquid dynamics." (1, p. IV-4)

Prior to the rocket flight, the samples were heated to well above the solidus (see Reference (1) or Reference (2) for time/temperature profiles of the experiment). At the time of the rocket launch, the sample temperatures ranged from 735 ^{o}C to 860

^{o}C. During the rocket flight, the samples in the first cavity were subjected to a high thermal gradient and the samples in the second cavity were subjected to a low thermal gradient. The samples were molten throughout the low-gravity phase of the rocket flight. Solidification was initiated prior to the reentry period by introducing a He gas flow along the outside of the cavities. Similar samples were processed on Earth for comparison.

Post-flight examination of the samples was achieved via (1) gamma-ray and metallographic (light metallography, SEM) techniques as well as (2) thermal analysis techniques. (A discussion of the thermal analysis is provided in Reference (1).) It was reported that the main result of the research "...lies in the preservation of a certain degree of dispersion of the indium primary phase. This result is radically different from those already obtained under microgravity conditions on the same system and compositions [e.g., see Löhberg, SPAR 2 (this chapter)]." (2, p. 252)

The main reasons for the above result were reported to be:

(1) capillarity factors: (a) differential wetting of Al and In on the silicon carbide cavity surface and (b) capillary convection due to thermal gradients and concentration gradients and

(2) solidification factors: (a) interaction between solidification front and second phase material and (b) coalescence of second phase globules.

It was further reported that the presence of free volumes created difficulties in interpreting the results, as expected (see Reference (1) or Reference (2) for detailed discussion of results).

Analysis/Results of each of the four flight samples and similarly processed ground based samples are presented in detail in Reference (1).

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Monotectic Compositions, Hypermonotectic Compositions, Metallic Matrix, Phase Separation, Melt and Solidification, Directional Solidification, Interface Physics, Solidification Front Physics, Homogeneous Dispersion, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Particle Coalescence, Drop Coalescence, Segregation, Free Surface, Surface Tension, Thermal Gradient, Solutal Gradients, Wetting, Wetting of

Container, Capillary Flow, Capillary Forces, Thermocapillary Convection, Marangoni Convection, Solid/Liquid Interface, Quench Process

Number of Samples: four
Sample Materials: three Al-32.08 wt.% In samples, one Al-16.0 wt.% In sample
(Al*In*)
Container Materials: silicon carbide
Si*C*

Experiment/Material Applications:
The specific reason why these Al-In alloys were selected for the experiments was not detailed in the available publications.

References/Applicable Publications:

(1) Potard, C.: SPAR IX Experiments 76-51/1 and 76-51/2 Directional Solidification of Immiscible Aluminum-Indium Alloys. In Space Processing Applications Rocket (SPAR) Project, SPAR IX Final Report, NASA TM-82549, pp. IV-1 - VI-79, January 1984. (post-flight)

(2) Potard, C.: Structures of Immiscible Alloys Solidified Under Microgravity Conditions. Acta Astronautica, Vol. 9, No. 4, pp. 245-254. (post-flight)

(3) Directional Solidification of Al-In Alloys in Microgravity: Results of the Basic Preparatory Investigations. AIAA 17th Aerospace Science Meeting, New Orleans, 1978, pp. 1-8. (preflight)

(4) Input received from Experiment Investigator, July 1989 and August 1993.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 5
Launch Date/Expt. Date: April 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 02-1 (large-chamber furnace and acoustic mixer). (The mixer was designed to operate within previously existing furnace hardware.)
Builder of Processing Facility: Acoustic Mixer: Battelle Institute, Frankfurt, Germany; Furnace: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Germany

Experiment:
Acoustic Mixing

Previous low-gravity research concerning the processing of immiscible alloys (e.g., see Fredriksson, TEXUS 2 (this chapter)) revealed that the solidification behavior of these systems was more complicated than originally anticipated. Sedimentation and buoyancy, for example (gravity-dependent phenomena) were not the only factors contributing to the separation of alloy constituents. Wetting and thermocapillary effects also limited the stability of the alloy systems.

This TEXUS 5 experiment was the first in a series of investigations designed by Heide and/or Langbein et al. to study the behavior of immiscible systems under low-gravity conditions.

An acoustic mixer, used to ultrasonically mix metallic melts, was developed for the experiment. The mixer allowed low-temperature processing of the immiscible material: the two components were heated to just above their respected melt temperatures. (Previously, alloys had been heated above the miscibility gap to mix the components.) Thus, the benefits of acoustic mixing included (1) a reduction in experiment power requirements and (2) a shortened sample cooling time. These benefits were important because of the short low-gravity period (approximately 6 minutes) available during the TEXUS sounding rocket mission.

Reportedly, the objectives of the investigation were to (1) functionally test the acoustic mixer under low-gravity conditions, (2) produce a fine dispersion alloy from a binary system exhibiting a liquid-phase miscibility gap, and (3) study the particle growth in a finely-dispersed metal-melt emulsion.

Prior to launch, a Zn-5 wt.% Pb sample was placed in a metal cartridge. Two thermocouples, configured at the bottom of the first- and second-thirds of the outside of the crucible (T1 and T2, respectively) were used to monitor temperature. The mixing system consisted of a piezoelectrically excited, stepped horn transducer which was mounted to the furnace structure. The acoustic energy radiates into the molten material via a mixing tool. "The front face cooler... [was]... fixed to the [sample] cartridge and in direct contact with the melt. The tip of the mixing tool... [was]... sealed against the mounting structure by a metal diaphragm to confine the metal melt to the cartridge. [The] cartridge and diaphragm... [were]... CVD-coated with TiN or covered with flexible graphite foil to avoid inter-metallic alloying with the contacting melt." (1, p. 100)

A functional test of the hardware was conducted between 400 and 350 seconds before launch. Then, just prior to launch, the Pb-Zn alloy was melted using the TEM 02 large chamber furnace equipped with the acoustic mixer.

Sixty seconds after launch, the mixer was automatically initiated. The sample temperature was between 474 and 484 ^{o}C) at this time. At 150 seconds after launch, directional solidification was initiated by He blast cooling. Thermocouple T1 indicated that the temperature at this location was 418 ^{o}C (solidification temperature) at 255 seconds after launch. At this time "...the ultrasonic mixing was interrupted to allow undisturbed coagulation of the Pb-Zn emulsion within the middle zone of the sample." (1, p. 101) The mixer was then switched back on when the solidification front reached T2 (309 seconds after launch). The mixer was switched off when the temperature at T2 reached 390 ^{o}C (380 seconds after launch). On Earth, reference samples were similarly processed for comparison.

Post-flight examination of the Pb-Zn sample indicated that the performance of the acoustic mixing system was satisfactory and the metal melt was emulsified. Specifically, "...the coalescence of the inclusions during the directional solidification of a binary alloy with miscibility gap in the liquid state can be counteracted by continuously dispersing them with an acoustic mixer." (4, p. 268) As expected, at the time the acoustic mixer was shut off, the low-gravity sample exhibited weaker coagulation than observed in the 1-g reference sample. In both samples coagulation resulted in an increase in the lead particle diameter when the mixer was swithched off. However, the diameters of the lead particles in the low-gravity sample were smaller than those of the 1-g sample. This difference was attributed to the weaker coagulation in the low-gravity processed material.

Since the relatively low lead content (5 wt.% Pb = 3 vol.% Pb) ensured emulsification and the mixing system was successfully tested, it was decided that during the next low-gravity experiment (see Heide, TEXUS 8 (this chapter)) a sample material containing a larger volume percent of the minority phase would be processed.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Metallic Matrix, Phase Separation, Melt and Solidification, Directional Solidification, Acoustic Mixing, Sedimentation, Buoyancy Effects, Separation of Components, Liquid/Liquid Interface, Liquid/Liquid Dispersion, Homogeneous Dispersion, Stability of Dispersions, Emulsion, Inclusions, Coagulation, Particle Coalescence, Particle Size Distribution, Particle Growth, Wetting, Surface Tension, Thermal Gradient, Thermocapillary Flow, Thermocapillary Convection, Marangoni Convection, Solid/Liquid Interface, Material Interaction with Containment Facility, Coated Surfaces, Quench Process

Number of Samples: one
Sample Materials: immiscible alloy: 5 wt.% Pb, 95 wt.% Zn
(Pb*Zn*)
Container Materials: metallic cartridge and diaphram coated with with TiN or flexible graphite
(Ti*N*, C*)

Experiment/Material Applications:
The specific reason why the Pb-Zn alloy was selected for this experiment was not detailed in available publications.

See also Fredriksson, TEXUS 2.

References/Applicable Publications:
(1) Clancy, P. F., Heide, W., and Langbein, D.: Sounding Rocket Flight Test of an Acoustic Mixer by Manufacture of a Lead-Zinc Emulsion Alloy in Microgravity. In Proceedings of the 4th European Symposium on Material Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, pp. 99-104. (post-flight)

(2) Clancy, P. F. and Heide, W.: Acoustic Mixing of an Immiscible Alloy (Pb-Zn) in Microgravity. In The Effect of Gravity on the Solidification of Immiscible Alloys, Proceedings of an RIT/ESA/SSC Workshop, Järva, Krog, Sweden, January 18-20, 1984, pp. 73-77. (post-flight)

(3) Acoustic Mixing. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 266-267. (post-flight)

(4) Solidification of Immiscible Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 268-269. (post-flight)

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Co-Investigator(s): Unknown
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 7
Launch Date/Expt. Date: May 1983
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-5
Builder of Processing Facility: Unknown

Experiment:
Separation of Transparent Liquids

This TEXUS 7 experiment was the second in a series of investigations designed by Heide and/or Langbein et al. to study the behavior of immiscible systems under low-gravity conditions (see Heide, TEXUS 5). The specific objective of the experiment was to examine the nucleation, growth, transport, and coalescence of minority phase droplets during directional cooling of a transparent, immiscible system.

The TEXUS Experiment Module TEM 06-5 was used for the experiment. The module contained a 40 mm x 20 mm x 10 mm aluminum block with two observation windows (the experiment cell). The observation windows allowed the experiment process to be filmed during the low-gravity mission. The cell was filled with a liquid consisting of 35% cyclohexane and 65% methanol. Two thermocouples, one at the top of the cell and one at the bottom of the cell were used to achieve thermal control of the sample liquid. In addition, a thermocouple on the left side of the cell and a thermocouple on the right side of the cell were used to monitor the fluid temperature.

One hour prior to launch, resistance heaters, attached to the top and bottom plates of the block, heated the sample liquid to 50 ^{o}C. (The mixture's critical temperature is 45.6 ^{o}C.) Full mixing of the components resulted. Once low-gravity conditions had been achieved (approximately 70 seconds after launch), the lower side of the liquid cell was cooled to 10 ^{o}C. (The lower heater was connected to a cooling plate such that, "...30 [seconds was] sufficient for cooling the bottom of the cell by 40 ^{o}C." (1, p. 28)) <Note: It appears that under the chosen thermal conditions, the liquid mixture does not solidify but a cooling front can be observed.>

Post-flight examination of the documenting film revealed the propagation of the cooling front and the growth of the cyclohexane particles behind the cooling front. (This migration was referred to as a "fog front" of cyclohexane particles.) Approximately 30 seconds after cooling was initiated, strong migration of the cyclohexane droplets towards the cooling front was observed. Reportedly, the migration of the droplets was caused by Marangoni convection. The Marangoni convection was attributed to the thermal and solutal gradients which existed behind the cooling front. Once the droplets reached the cooling front, the migration was halted since the liquid above the front was at a temperature of 50 ^{o}C (and thus no thermal gradient existed to drive the Marangoni convection). It was reported that the results from this experiment fit well with the theoretical predictions (see Reference (3) for discussions concerning the theoretical treatment).

A reference experiment was performed on Earth for comparison. The fluid behavior of the ground sample was similar to that of the rocket sample during the first 35 seconds. However, the cyclohexane drops, now driven by gravity-induced buoyancy forces, continued to move into the liquid. Once in the liquid, where the two fluids were still miscible, the droplets shrank and vanished after a few seconds.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Fluids, Binary Systems, Model Materials, Transparent Liquids, Phase Separation, Directional Solidification, Thermal Gradient, Solutal Gradients, Liquid Mixing, Emulsion, Dispersion, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Drops, Drop Coalescence, Drop Migration, Particle Growth, Particle Transport, Nucleation, Segregation, Buoyancy Effects, Thermocapillary Convection, Buoyancy-Driven Convection, Marangoni Convection, Marangoni Movement of Droplets, Solidification Front Physics, Solid/Liquid Interface

Number of Samples: one
Sample Materials: binary liquid: 35% cyclohexane and 65% methanol
Container Materials: aluminum
(Al*)

Experiment/Material Applications:
The cyclohexane/methanol mixture used in this experiment represents a model system of immiscible materials. The liquid is transparent and permitted visualization of drop movement.

References/Applicable Publications:

(1) Langbein, D. and Heide, W: The Separation of Liquids Due to Marangoni Convection. Adv. Space Res., Vol. 4, No. 5, 1984, pp. 27-36. (post-flight)

(2) Langbein, D. and Heide, W.: Entmischung von Flüssigkeiten aufgrund von Grenz-flächenkonvektion. ZFW, Vol. 8, 1984, pp. 192-199. (in German)

(3) Langbein, D.: On the Separation of Alloys Exhibiting a Miscibility Gap. In Proc. Workshop on Effect of Gravity on Solidification of Immiscible Alloys, Stockholm, January 18-20, 1984, ESA SP-219, March 1984. (theoretical analysis)

(4) Langbein, D. and Heide, W.: Study of Convective Mechanisms Under Microgravity Conditions. Adv. Space Res., Vol. 6, No. 5, pp. 5-17, 1986. (TEXUS 7 and 9)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 8
Launch Date/Expt. Date: May 1983
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 02-1 (furnace with acoustic mixer)
Builder of Processing Facility: Acoustic Mixer: Battelle Institute, Frankfurt, Germany; Furnace: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Germany

Experiment:
Acoustic Mixing/Solidification of Immiscible Alloys

This TEXUS 8 experiment was the third in a series of investigations designed by Heide and/or Langbein et al. to study the behavior of immiscible systems under low-gravity conditions (see Heide, TEXUS 5, TEXUS 7). The major objective of the TEXUS 8 experiment was the same as that for the earlier TEXUS 5 experiment: to obtain a fine dispersion in an immiscible material system by acoustic mixing. In contrast to the TEXUS 5 experiment, the TEXUS 8 experiment employed an alloy which had a critical concentration of the minority component.

Prior to the mission, a Zn-15 wt.% Pb sample was prepared. The higher Pb content (higher than the TEXUS 5 content) was used because (1) solidifying a Zn-15 wt.% Pb alloy on Earth is problematic, (2) such an alloy is more interesting from a technical point of view, and (3) an alloy with this large of a volume percent of minority phase is likely to provide information concerning active segregation mechanisms.

The TEXUS Experiment Module TEM 02-1, equipped with an acoustic mixer, was used for the study. The experimental setup and procedure was the same as that described under Heide, TEXUS 5.

Post-flight examination of the low-gravity sample indicated that the section processed with acoustic mixing contained Pb particles with diameters of up to 50 microns. When the acoustic mixer was switched off, the Pb particle size increased to between 200 and 300 microns. In a similarly processed ground-based sample, the particle size increased from 120 to 150 microns for the respective sections. It was also reported that, for both 1-g and low-g samples, the Pb volume content in the sections solidified with acoustic mixing was lower than the initial lead content.

"It is clear from these results that for the case of an immiscible alloy such as Pb-Zn with a critical concentration of the minor component (Pb)... [that]... in the absence of mixing, rapid and extreme coagulation occurs. In this case [acoustic] mixing can be used in microgravity conditions to produce a fine dispersion with results better than those achievable on the ground even with mixing and is a necessary technique to prevent rapid coagulation of a critical composition in microgravity." (1, p. 77)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Metallic Matrix, Phase Separation, Melt and Solidification, Acoustic Mixing, Sedimentation, Segregation, Buoyancy Effects, Separation of Components, Minority Phase, Liquid/Liquid Interface, Liquid/Liquid Dispersion, Homogeneous Dispersion, Stability of Dispersions, Emulsion, Coagulation, Particle Size Distribution, Particle Growth, Solid/Liquid Interface, Coated Surfaces

Number of Samples: one
Sample Materials: immiscible alloy: Zn-15 wt.% Pb (Zn*Pb*)
Container Materials: See Heide, TEXUS 5

Experiment/Material Applications:
See **Experiment** summary (above).

See also Fredriksson, TEXUS 2, "Segregation Phenomena in Immiscible Alloys: Zn-Bi" (this chapter).

References/Applicable Publications:
(1) Clancy, P. F. and Heide, W.: Acoustic Mixing of an Immiscible Alloy (Pb-Zn) in Microgravity. In The Effect of Gravity on the Solidification of Immiscible Alloys, Proceedings of an RIT/ESA/SSC Workshop, Järva Krog, Sweden, January 18-20, 1984, pp. 73-77. (post-flight; discusses TEXUS 5 and TEXUS 8 experiments)

(2) Clancy, P. F., Heide, W., and Langbein, D.: Sounding-Rocket Flight Test of an Acoustic Mixer by Manufacture of a Lead-Zinc Emulsion Alloy in Microgravity. In Proceedings of the 4th European Symposium on Materials Sciences under Microgravity, Madrid, Spain, April 5-8, 1983, pp. 99-104. (preflight; TEXUS 5 results and justification for TEXUS 8 sample material)

(3) Solidification of Immiscible Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 268-269. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 9
Launch Date/Expt. Date: May 1984
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-5
Builder of Processing Facility: Unknown

Experiment:
Separation of Transparent Liquids

This TEXUS 9 experiment was the fourth in a series of investigations designed by Heide and/or Langbein et al. to study the behavior of immiscible systems under low-gravity conditions (see Heide, TEXUS 5, TEXUS 7, TEXUS 8). The specific objectives of the experiment were to (1) observe the nucleation, growth, and Marangoni migration of minority phase droplets during directional solidification of a transparent, immiscible system and (2) investigate the effects of a moving solidification front on the Marangoni migration.

The experiment apparatus and procedure were essentially the same as those described under Heide, TEXUS 7. The major difference between the two flight experiments was that TEXUS 9 (1) employed a 5 wt.% methanol/95 wt.% cyclohexane mixture and (2) this mixture was sufficiently cooled to create a solidification front. (The solidification temperature of cyclohexane is +6 ^{o}C.)

Prior to launch, the experiment cell was heated to 50 ^{o}C (well above the liquid phase miscibility gap of this material system). Once low-gravity conditions had been achieved, the bottom plate of the experiment cell was cooled to -5 ^{o}C creating a solidification front. The experiment was recorded with a 16 mm cine camera.

Post-flight analysis of the documenting film revealed that the initial behavior of the TEXUS 9 system was similar to the TEXUS 7 experiment: "...there is the penetration of the cooling and fog front. However, about 10 s after... [the cooling is initiated] a second, darker fog front moves upwards. It is faster than the first one and passes it after about 18 s. It turns out to be a front of methanol droplets, which are undergoing collective Marangoni migration. Again, after about 30 s larger methanol droplets migrate from the bottom to the cooling front.... After 36 s, when the bottom of the cell has reached 6 ^{o}C, i.e. when

solidification of cyclohexane starts, the distribution of the methanol droplets becomes more uniform, their average size decreases. The solidification front hinders a preferred nucleation at bottom roughness." (2, p. 9)

When the TEXUS 9 results were compared to those from TEXUS 7, it was reported that coagulation of methanol droplets in a cyclohexane matrix (TEXUS 9) was much faster than coagulation of cyclohexane droplets in a methanol matrix (TEXUS 7). This result occurred despite the fact that both systems have the same increase in interface energy. It was also reported that convective rolls formed during the TEXUS 9 experiment after about 3 minutes. These rolls "...can be ascribed to a correlation between the growth front of cyclohexane and the nucleation and Marangoni migration of methanol droplets." (2, p. 9) <Note: No mention of convection rolls were reported for the TEXUS 7 experiment.>

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Fluids, Binary Systems, Phase Separation, Model Materials, Transparent Liquids, Directional Solidification, Thermal Gradient, Solutal Gradients, Minority Phase, Emulsion, Dispersion, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Drops, Coagulation, Drop Migration, Particle Growth, Particle Transport, Droplet Size, Particle Size Distribution, Nucleation, Segregation, Buoyancy Effects, Marangoni Convection, Marangoni Movement of Droplets, Interfacial Energy, Solidification Front Physics, Solid/Liquid Interface

Number of Samples: one
Sample Materials: binary liquid: 95 wt.% cyclohexane and 5 wt.% methanol
Container Materials: aluminum
(Al*)

Experiment/Material Applications:
See Heide, TEXUS 7.

References/Applicable Publications:

(1) Langbein, D. and Heide, W.: The Separation of Liquids Due to Marangoni Convection. Advances in Space Research, Vol. 4, Number 5, 1984, pp. 27-36. (post-flight; discusses results from TEXUS 7 and TEXUS 9 experiments)

(2) Langbein, D. and Heide, W.: Study of Convective Mechanisms Under Microgravity Conditions. Adv. Space Res., Vol. 6, No. 5, 1986, pp. 5-17. (post-flight TEXUS 7, 9 and D1)

(3) Marangoni Transport of Droplets at a Solidification Front. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 272-273. (post-flight)

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Experiment Origin: USA
Mission: STS Launch #4, STS-004 (STS OFT-4, Columbia)
Launch Date/Expt. Date: June 1982
Launched From: NASA Kennedy Space Center, Florida
Payload Type: College Student Experiment
NASA Get Away Special (GAS) Canister G-001
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-001: Utah State University, Logan, Utah/R. Gilbert Moore
Processing Facility: Hot wire cell/pump-heater assembly
Builder of Processing Facility: Designed by: Principal Investigator R. Laher; machined off-campus, Logan Utah

Experiment:
Thermal Conductivity of a Binary Heterogeneous Mixture (Experiment Number 9-P)

On Earth, accurate measurements of the thermal conductivity of an immiscible liquid are hindered (in part) by (1) fluid heat losses attributed to convective flow and (2) separation of differing density constituents of the mixture. In a low-gravity environment, such heat losses and fluid demixing should be reduced allowing a more accurate measurement of the thermal parameter.

This experiment was one of ten investigations housed within the G-001 Get Away Special (GAS) canister during STS-004. (Four other experiments (of the ten) were applicable to this data base (see Alford, STS-004 (Chapter 18); Dalley, STS-004 (Chapter 5); Elwell, STS-004 (Chapter 12); Thomas, T. L., STS-004 (Chapter 14)).) The specific objective of the experiment was to measure the thermal conductivity of a binary heterogeneous mixture.

The experimental setup included (1) an emulsification device to mix the test liquids (crude oil and water) and (2) a hot wire liquid receiver cell (a hollow cylinder with a nichrome heater positioned along its long axis).

The expected, low-gravity operational scenario consisted of (1) emulsifying the oil and water components, (2) pumping the emulsified mixture into the hot wire cell, (3) applying a voltage across the heater wire to induce a radial thermal gradient in the fluid, (4) allowing a sufficient amount of time for the thermal field to achieve a steady state, and (5) measuring the temperature distribution.

Reportedly, the resultant thermal data was to be used to calculate the thermal conductivity of the mixture. However, post-flight analysis of the experimental payload revealed that the water in the experiment froze before the payload could be activated. Consequently, the experiment was aborted. The principal investigator commented that perhaps the most important results of all were (1) the experience and knowledge acquired, and (2) the personal enrichment from working with many dedicated and talented individuals involved in bringing about payload G-001.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Fluids, Phase Separation, Binary Systems, Thermal Conductivity Measurements, Heat Transfer, Thermal Gradient, Thermal Distribution, Buoyancy-Driven Convection, Separation of Components, Density Difference, Liquid Mixing, Emulsion, Dispersion, Liquid/Liquid Dispersion, Liquid Transfer, Electric Field, Thermal Environment More Extreme Than Predicted, Freezing, Hot-Wire Technique, Contained Fluids, Liquid Reservoir

Number of Samples: one
Sample Materials: mixture of Louisiana crude oil & water
Container Materials: aluminum
(Al*)

Experiment/Material Applications:
Data resulting from research such as this could be used to validate existing theoretical treatments of the thermal conductivity of a binary heterogeneous mixture.

References/Applicable Publications:

(1) Yoel, D., Walker, S., Elwell, J. and Moore, G.: The First Getaway Special - How it was Done. Spaceworld, May 1983, pp. 9-16. (post-flight)

(2) STS-4 Fourth Space Shuttle Mission, NASA Press Kit, June 1982, p. 62. (preflight)

(3) Yoel, D. W.: Payload Integration of a Get Away Special Canister. American Institute of Aeronautics and Astronautics, Annual Meeting and Technical Display on Frontiers of Achievement, Long Beach, California, May 12-14, 1981, 5 pp. (preflight)

(4) The STS-4 Getaway Special. NASA Report PB82-10223, May 20, 1982. (preflight)

(5) Cargo Systems Manual: GAS STS-4, May 20, 1988, JSC-17645, pp. 4-1 - 4-4. (preflight; very short description)

(6) Overbye, D.: The Getaway Kids Shuttle Into History. Discover, September 1982. (post-flight)

(7) Yoel, D. W.: Analysis of the First Getaway Special Space Shuttle Payload. Thesis for M.S. in Physics, Utah State University, Logan, Utah, 1984.

(8) Moore, R. G.: Educational Implications of Getaway Special Payload Number One. IAF-81-293, XXXIInd International Astronautical Federation Congress, Rome, September 6, 1981.

(9) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(10) Input received from Principal Investigator R. Laher, August 1989.

(11) Transcripts of press conference at NASA MSFC with G-001 student experimenters and sponsors, NASA, May 20, 1982.

(12) "Get Away Special," NASA News, NASA MSFC, June 7, 1982.

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #5, STS-005 (STS 31-A, Columbia)
Launch Date/Expt. Date: November 1982
Launched From: NASA Kennedy Space Center, Florida
Payload Type: West German Get Away Special (GAS) MAUS Canister DG-205 (Also designated as NASA Get Away Special (GAS) Canister G-026)
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of DG-205/G-026: The German Ministry of Research and Technology (BMFT)/Messerschmitt-Boelkow-Blohm (MBB-ERNO)
Processing Facility: Radiation transparent thermostat (oven) with X-ray unit. Variable cooling rates of the sample were possible via the use of an air fan.
Builder of Processing Facility: DFVLR, Institute of Space Simulation, Cologne, Germany

Experiment:
Stability of Metallic Dispersions (DG-205)

Above a certain temperature (the consulate temperature), a specific combination of gallium and mercury illustrates solubility in the liquid state. When such a Hg-Ga system is processed on Earth, the molten mercury rapidly separates from the molten gallium because of a large density difference between the alloying components. In contrast, when such a system is processed in space, there is a reduction of the gravity-driven forces (sedimentation and buoyancy) which separate the metals. Thus, it was anticipated that in a low-gravity environment (1) a more homogeneous dispersion of the mercury droplets in the gallium could be attained and (2) gravity-independent forces responsible for the dispersion could be more closely investigated. It was also surmised that if X-rays of the sample could be used to record the appearance of the liquid metal shortly before or during solidification, the physical processes governing the resultant product might be more clearly defined.

This STS-005 Get Away Special (GAS) metals-mixing experiment was the first in a series of investigations designed by Otto to study the stability of metallic dispersions under low-gravity conditions. The major objective of the investigation was to process a

Ga-Hg sample in a transparent heater while simultaneously penetrating the liquid alloy with periodic X-rays. Such X-ray radiography would provide a real-time examination of the metal during different stages of the experiment. The processing/X-ray examination would permit the investigation of (1) the dissolution process of the Ga-Hg system above the consulate temperature and (2) the time-dependent stability of the dispersion (composed of mercury droplets in gallium).

<Note: Reportedly, another major objective of the experiment was testing the "function" of the MAUS standard system. Although this objective was not further explained, it is thought that testing the function may have implied determining the success and practicality of the West German Get Away Special containers. (Details of the MAUS system can be located in Reference (5).)>

During the mission, the single sample (80 vol.% Ga - 20 vol.% Hg) was to be processed. Because the sample "...could be recycled into its starting conditions by repeated thermal treatment...," (1, p. 104) the thermal cycling was to be performed during the 3 days of planned experiment time.

Post-flight analysis of the payload indicated that the experiment was not activated. Reportedly, "A failure analysis yielded that a leak in a silver-zinc electronic battery had developed during the several weeks of waiting time on ground. Because of no voltage conditions the electronics of the standard system could not be activated by the "on" signal given by the crew." (1, p. 104) The sample payload was reflown on the NASA structure OSTA-2 during the space shuttle STS-007 mission (see Otto, STS-007).

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Metals, Metallic Matrix, Phase Separation, Melt and Solidification, Drops, Drop Formation, Particle Growth, Nucleation, Dispersion, Dispersion Alloys, Dispersion Strengthening, Stability of Dispersions, Metallic Dispersion, Liquid/Liquid Dispersion, Homogeneous Dispersion, Liquid/Liquid Interface, Density Difference, Separation of Components, Sedimentation, Buoyancy Effects, Dissolution, Precipitation of Second Phase, Solid/Liquid Interface, X-Ray of Liquid Metal in Space, Air Fan, Sample Not Processed As Planned, Battery Drain, Battery Leakage

Number of Samples: one
Sample Materials: 80 vol.% gallium - 20 vol.% mercury (Ga*Hg*)

Container Materials: Teflon™

Experiment/Material Applications:
The lack of direct observation of the physical processes occurring in liquid metals, either just before or during solidification, hampers the study of physical phenomena within the system. Post examination of materials requires interpretation of the processes which occurred during the solidification, which is difficult since details of these intermediate stages are missing. The use of X-rays would allow real-time observation of these processes.

Understanding the precipitation process (including nucleation, growth and ripening) will lead to the improvement of dispersion strengthened materials.

References/Applicable Publications:

(1) Otto, G. H. and Baum, D. Material Sciences Experiments Under Microgravity Conditions With M*A*U*S. In NASA Goddard Space Flight Center's 1985 Get Away Special Experimenter's Symposium, October 8-9, 1985, pp. 101-108, NASA CP-2401. (post-flight)

(2) Cause of German Payload Failure Determined. Aviation Week and Space Technology, April 11, 1983. (post-flight)

(3) Moser, J. F.: Cargo Systems Manual: GAS STS-5. NASA JSC-17645 September 12, 1982, p. 4-1. (preflight)

(4) Otto, G. H.: The Behaviour of a Metallic Dispersion Under Microgravity Conditions. Proceedings of the 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191. (preflight)

(5) Baum, D., Otto, P., and Vits, P.: MAUS-A Flight Opportunity for Automated Experiments Under Microgravity Conditions. Acta Astronautica, Vol. 11, pp. 239-245, 1984.

(6) Baum, D., Stolze, H., and Vits, P.: Flight Data from MAUS Payloads. IAF Paper 84-137, 1984.

(7) Otto, G. H.: MAUS für Legierungen in der Schwerelosigkeit. Umschau, Vol. 82, p. 703, 1982. (preflight)

(8) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report # EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(9) Input received from Principal Investigator G. H. Otto, July 1989 and August 1993.

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: West German Get Away Special (GAS) MAUS Canisters DG-205I and DG-205II
Volume of Canisters: 5.0 cubic feet
Location of Canisters: STS Payload Bay (carried on the Office of Space and Terrestrial Applications (OSTA-2) pallet (a NASA experiment carrier))
Primary Developer/Sponsor of DG-205I, DG-205II: The German Ministry for Research and Technology (BMFT)/Messerschmitt-Boelkow-Blohm (MBB-ERNO)
Processing Facility: Radiation transparent thermostat (oven) with X-ray unit. Variable cooling rates of the sample were obtained via the use of an air fan. (This facility was similar to the processing facility configured on MAUS DG-205, STS-005, with no upgrades.)
Builder of Processing Facility: DFVLR, Institute of Space Simulation, Cologne, Germany

Experiment:
<u>The Stability of Metallic Dispersions (DG-205I and DG-205II)</u>

This STS-007 MAUS Get Away Special (GAS) metals-mixing experiment was the second in a series of investigations designed by Otto to study the stability of metallic dispersions under low-gravity conditions (see Otto, STS-005).

Two MAUS canisters onboard STS-007 (DG-205I and DG-205II) were dedicated to the dispersion experiment. Both of the canisters were mounted on the OSTA-2 carrier in the shuttle cargo bay. Reportedly, DG-205I and DG-205II had similar hardware configurations, but sample conditions and time-temperature profiles differed. For example, the Principal investigator noted that (1) the DG-205I processed a 80 vol.% Ga, 20 vol.% Hg sample, and that (2) the DG-205II processed a 83.8 vol.% Ga, 16.2 vol.% Hg sample.

The STS-007 experiment was similar to the earlier STS-005 investigation. The major objective of the investigation remained the same: to process a Ga-Hg sample in a transparent heater while simultaneously penetrating the liquid alloy with periodic X-rays.

Such X-ray radiography could provide a real-time examination of the metallic melt during different stages of the experiment. The processing/X-ray examination would permit the investigation of (1) the dissolution process of the Ga-Hg system above the consulate temperature and (2) the time-dependent stability of the dispersion (composed of mercury droplets in gallium).

Because a battery leak occurred in the previous STS-005 MAUS payload, the cause of the leak, a simple O-ring seal, was "corrected" for the STS-007 flight of the DG-205 canister.

During the mission, a single sample was processed in each of the MAUS canisters with three heating and cooling cycles. A reservoir, configured to compensate for volume expansion of the sample, was implemented to eliminate material free surfaces.

A document published prior to the launch of the experiment detailed an expected experiment cycle. First, the samples were to be heated to a temperature above the miscibility gap (220 $^{\circ}$C) and homogenized. Second, the samples were to be cooled into the miscibility gap (but not solidified) with a prescribed cooling rate. Third, the dispersion was to be held at a constant temperature. Fourth, because the samples "...could be recycled into... [their] starting conditions by repeated thermal treatment..." (4, p. 104) (by heating the sample to its homogenized state above the miscibility gap (220 $^{\circ}$C)), the thermal cycling was to be repeated during the 3 days of planned experiment time.

During the mission, "Different cooling rates of 30, 10 and 2 K/min were achieved by forced cooling with a fan, natural and programmed cooling respectively. In the actual experiment the cycle containing natural cooling was lost because of temporary problems with the film transport." (2, p. 44) The differing cooling rates permitted an examination of rate-dependent processes (precipitation and growth). The temperature hold at the miscibility gap permitted an examination of isothermal processes (droplet motion by residual gravity or droplet growth via Ostwald Ripening).

<Note: Although it is clear from Reference (3) that DG-205I was cooled into the miscibility gap at a rate of 30 K/min and that DG-205II was cooled into the miscibility gap at a rate of 1.7 K/min, details/results of other cooling cycles in each canister were not presented (although references indicated that four experiment cycles (total) were realized). Thus, it appears from Reference (2) that these other two cooling rates may have been related to the lost data referred to above (natural cooling 10 K/min).>

Post-flight analysis of the payload indicated that the experiment was successful and "...yielded the first X-ray photos from a metallic dispersion cooling a homogeneous solution into the miscibility gap." (4, p. 104)

The following observations were reported:

"- [Homogenization] appears to be completed after 4 hours at 190 $^{\circ}$C. This can be concluded from the constant grey [sic] scale value of the sample when measuring across the X-ray film. In the [Earth] laboratory at least 8 hours are needed for worst case conditions when the heavier mercury is on the bottom of the container.

[<Note: Reference (3) indicated that in DG-205I, the homogenized state was achieved after a diffusion time of 24 hours at 190 $^{\circ}$C when a cooling rate of 30 K/min was employed. Further, Reference (5) indicated that low-g homogenizations of gallium and mercury by diffusion (cooling rate not specified) were achieved in less than 1 hour. The Principal Investigator addressed these "inconsistencies" in reporting by explaining that "...at the time when the experiment was designed the... [homogenization] durations were very much in question because the convective contribution to the diffusion coefficient in the liquid state was not known. Therefore, a conservative... [homogenization] time of 24 hours [prior to cooling] was chosen in order to be on the safe side. After the experiment it turned out that this time was sufficient. However, depending on the dispersed state of the sample... [homogenization] was achieved in less than 1 hour.>]

"- When cooling the sample in to the miscibility gap with a rate of 30 K/min the precipitation of the Hg-droplets occurs rapidly. However, no finely dispersed state with a particle size of about 0.3 diameter ([the] resolution limit of the X-ray photos) can be observed. Hg-droplets seem to be generated by heterogeneous nucleation at the gallium surface. Droplets seem to be stationary once they achieve the visibility limit and do not show any blurring movement despite the exposure time of 20 s.

"- Supercooling of the melt appears small and if present should be less than 20 $^{\circ}$C.

"- When cooling into the gap the growth of precipitated droplets [in the low-g environment] is rather fast.... Within one minute (30 K into the gap) the particles have already grown to an average diameter of 0.8 mm. Anticipating growth by diffusion only, the diameters increase too fast by at least a factor of five. Other processes like convective material transport or coalescence are likely to contribute to growth....

[<Note: Reportedly, concentration gradients at the interface were sources of the convective material transport (see Reference (10)). Acceleration levels on the shuttle were mentioned below.>]

"The housekeeping systems also provided information about the payload from which the acceleration data taken over a period of three days are the most interesting.... Crew activities and activation of the robotic arm can be seen clearly on the record. It should be stated that the g-sensitive runs of the X-ray experiment were programmed to happen during the sleeping time of the crew." (4, pp. 104-105)

"- Movement of the droplets due to residual acceleration over a period of two hours cannot be observed. Therefore, it is concluded that the mercury precipitated or rapidly migrated to the... [gallium/teflon(container) interface] of the sample where it became stationary." (2, p. 45)

It was concluded that a homogeneous dispersion of a Hg-rich phase was not achieved during the experiment. Instead, there was a tendency for the mercury to coagulate into droplets.

"In the sample that was cooled with a fast rate the process of precipitation and nucleation of droplets occurred very quickly. In the photographs one sees the sudden appearance of droplets, heterogeneously distributed in location as well as size. Minimum size detectable is 0,2 mm diameter. The size of the droplet grows with further cooling. The rate of growth of the droplets is faster than one would expect by diffusion alone. Supposedly convection in microgravity has played a part.

"Cooling into the miscibility gap with the considerably slower rate of 1,7 K/min the growth of the precipitated droplets is slower and can be explained by the amount of mercury being available. This amount is governed by the phase diagram and is actually less than could be consumed by a purely diffusive process." (3, p. 388)

Many other interesting conclusions are detailed in References (3) and (10).

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Metals, Metallic Matrix, Phase Separation, Melt and Solidification, Drops, Drop Formation, Drop Migration, Drop Coalescence, Droplet Size, Particle Growth, Nucleation, Heterogeneous Nucleation, Dispersion, Dispersion Alloys, Dispersion Strengthening, Stability of Dispersions, Metallic Dispersion, Liquid/Liquid Dispersion, Homogeneous Dispersion, Liquid/Liquid Interface, Ostwald Ripening, Coarsening, Coagulation, Density Difference, Separation of Components, Sedimentation, Stokes Sedimentation, Buoyancy Effects, Dissolution, Precipitation of Second Phase, Wetting, Solutal Gradients, Marangoni Convection, Surface Tension-Driven Convection, Diffusion, Diffusive Mass Transfer, Solid/Liquid Interface, Solidification Rate, Radiative Cooling, Air Fan, Supercooling, Volume Expansion, Volume Compensation, Free Surface Elimination, X-Ray of Liquid Metal in Space, Acceleration Effects, Acceleration Measurement

Number of Samples: Two samples (one on DG-205I and one on DG-205II). Multiple runs were performed on each of these samples.
Sample Materials: DG-205I: 80 vol.% gallium - 20 vol.% mercury; DG-205II: 83.3 vol.% gallium - 16.2 vol.% mercury
(Ga*Hg*)
Container Materials: Teflon™

Experiment/Material Applications:
The processes of droplet nucleation, growth and wetting in low-gravity have to be understood before dispersion-strengthened alloys can be prepared. Coalescence of liquid droplets will lead to fast coarsening of samples.

See also Otto, STS-005.

References/Applicable Publications:
(1) Otto, G. H.: The Behavior Of a Metallic Dispersion Under Microgravity Conditions. In ESA 4th European Symposium On Material Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, Publication ESA SP-191, pp. 63-69. (preflight)

(2) Otto, G. H.: First Results of a MAUS Experiment To Investigate the Stability of a Metallic Dispersion. Workshop on Effect of Gravity on Solidification of Immiscible Alloys, Stockholm, January 18-20, 1984, ESA SP-219, pp. 43-46. (post-flight)

(3) Otto, G. H.: Stability of Metallic Dispersions. Proceedings of the 5th European Symposium on Material Science under Microgravity, Schloss-Elmau, November 5-7, 1984, pp. 379-388, ESA SP-222. (post-flight)

(4) Otto, G. H. and Baum D.: Materials Sciences Experiments Under Microgravity Conditions with M*A*U*S. In NASA Goddard Space Flight Center, The 1985 Get Away Special Experimenter's Symposium, pp. 101-108. (post-flight)

(5) Otto, G. H.: Experimental Results from Automated MAUS Payloads. IAF Paper 88-351 (1988). (post-flight)

(6) Otto, G. H.: MAUS-Nuklasten für Space Shuttle. Umschau, Vol. 83, pp. 394-395. (1983) (preflight)

(7) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(8) Input received from Principal Investigator G. H. Otto, July 1989 and August 1993.

(9) NASA STS-007 Press Kit, p. 50. (preflight)

(10) Otto, G. H. and Frohberg, G.: Droplet Dissolution Kinetics in the Miscibility Ga of Ga-Hg: Comparison of Microgravity Results with a Computer Simulation. In Proceedings of the 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, February 1987, pp. 335-360. (post-flight)

(11) Baum, D., Stolze, H., and Vits, P.: First Flight Data from MAUS Payloads on STS 7 and STS 11. 35th Congress of the International Astronautical Federation, October 7-13, 1984, Lausanne, Switzerland, 11 pp. (post-flight)

(12) Baum, D., Otto, G., and Vits, P.: MAUS-A Flight Opportunity for Automated Experiments Under Microgravity Conditions. Acta Astronautica, Vol. 11, No. 3-4, pp. 239-245, 1984. (no discussion of experiment results)

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Co-Investigator(s): None
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Experiment Origin: Great Britain
Mission: TEXUS 7
Launch Date/Expt. Date: May 1983
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Three mirror furnaces (designated as MF 4, MF 5, and MF 6) located in the Swedish TEXUS Experiment Module
Builder of Processing Facility: Unknown, Probably the Swedish Space Corporation, Solna, Sweden

Experiment:
Solidification of Al-Pb Alloys Under Microgravity

During the solidification of metallic alloys on Earth (1) the stability of an emulsion is hampered by gravity-induced sedimentation effects and (2) the role of surface energy on agglomeration and solidification front inclusion is masked by the overwhelming gravity forces.

This TEXUS 7 experiment was the first in a series of investigations designed by Caton and/or Goodhew et al. to study the stability of a metallic dispersion. The specific objective of the experiment was to study various dispersions of lead in liquid aluminum and to attempt to (1) increase emulsion stability and (2) determine the importance of surface energy in the system.

Prior to the mission, "...three aluminum samples containing lead in the range 6-8 wt.% (monotectic temperature = 660 $^{\circ}$C) were prepared..." (3, p. 264) such that they would exhibit a fine dispersion when processed.

During the low-gravity phase of the rocket, the samples were melted in three mirror furnaces. Reportedly, "Sample 1 was heated up to 950 $^{\circ}$C for 64 s; sample 2 was heated up to 950 $^{\circ}$C for 64 s and then maintained at 750°C for 115 s; sample 3 was heated up to 750 $^{\circ}$C for 64 s. All samples were rapidly solidified before the end of the microgravity period." (3, p. 264)

Post-flight, it was determined that:

(1) in flight sample 1 "...a fine dispersion had been produced and retained.... A denuded region on the surface of the specimen was observed, believed to result from variations in solidification front speed." (1, p. 83)

(2) "Sample 2 showed an unexpected segregation of large droplets in the last frozen liquid." (3, p. 264) and

(3) "Sample 3 also showed particles pushed in the direction of solidification but with an unexpected rim of larger droplets within the denuded region on the surface." (3, p. 264)

(4) "In view of the low solubility of Pb in Al at the monotectic point (1.5 wt%), and the rapid solidification of the samples, it was possible to... [analyze] the stability of the dispersion and its interaction with the solidification front as it swept through." (3, p. 264) <Note: No further discussion of this analysis was presented.>

It was further reported that similarly processed ground-based samples exhibited "...the expected agglomerations of lead in the direction of gravity." (3, p. 264)

No further information concerning this experiment could be located at this time.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Monotectic Compositions, Metals, Metallic Matrix, Dispersion Alloys, Dispersion Strengthening, Phase Separation, Melt and Solidification, Drops, Drop Formation, Droplet Agglomeration, Dispersion, Stability of Dispersions, Metallic Dispersion, Particle Dispersion, Droplet Dispersion, Liquid/Liquid Dispersion, Homogeneous Dispersion, Emulsion, Liquid/Liquid Interface, Density Difference, Separation of Components, Segregation, Sedimentation, Stokes Sedimentation, Buoyancy Effects, Surface Tension, Surface Energy, Interface Physics, Solid/Liquid Interface, Solidification Rate, Inclusion and/or Rejection of Particles, Solidification Front Physics, Superconductivity

Number of Samples: three
Materials: aluminum samples containing lead in the range 6-8 wt%.
(Al*Pb*)
Container Materials: unknown, appears to have been copper
(Cu*)

Experiment/Material Applications:
See Caton, Spacelab 1 (this chapter).

References/Applicable Publications:

(1) Hopkins, W. G.: Solidification of Al-Pb Alloys Under Microgravity in Texus-7, Preliminary Report. In ESA The Effect of Gravity on Immiscible Alloys, 1984, pp. 83-86. (post-flight)

(2) Input received from Experiment Investigator, November 1989.

(3) Immiscible Alloy System Al-Pb. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 264-265. (post-flight)

(4) Input received from Experiment Investigator T. W. Clyne, July 1993.

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Co-Investigator(s): None
Affiliation(s): (1,2) Fulmer Research Laboratories, Slough, Stoke Pages, Great Britain

Experiment Origin: Great Britain
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility (IHF) Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Metallic Emulsions Al-Pb 1ES309

When there is a significant density difference between the matrix melt and dispersion droplets in a metallic emulsion, the Earth-processed emulsion is affected by gravity-induced (1) segregation of the constituents and (2) agglomeration of the droplets. In the low-gravity environment however, such segregation and agglomeration should be reduced resulting in (1) increased emulsion stability and (2) improved component dispersion.

This experiment was the second in an series of investigations designed by Caton and/or Goodhew et al. to study the stability of a metallic dispersion (see Caton, TEXUS 7). (The rest of the experiments in the series are in Chapter 5 under Goodhew, TEXUS 12, TEXUS 14a, and TEXUS 14b.) The specific objective of the experiment was to solidify an aluminum melt containing a fine dispersion of lead droplets.

The expected solidification sequence was to be as follows: "...heat the aluminum-lead alloy samples into the single liquid region, allow sufficient time for complete... [homogenization], cool to predetermined temperatures in the two-liquid region, hold for a particle growth period and cool to solid state. Identical samples will be given a similar heat treatment cycle, except that the holding period in the two-liquid region will be omitted." (1, p. 91)

Documentation detailing the in-flight experiment performance or the post-flight experiment results could be located at this time. Investigator input indicated that no useful data were obtained.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Metals, Metallic Matrix, Phase Separation, Dispersion Alloys, Dispersion Strengthening, Melt and Solidification, Drops, Drop Formation, Droplet Agglomeration, Particle Growth, Stability of Dispersions, Metallic Dispersion, Particle Dispersion, Droplet Dispersion, Liquid/Liquid Dispersion, Homogeneous Dispersion, Emulsion, Liquid/Liquid Interface, Density Difference, Separation of Components, Segregation, Sedimentation, Stokes Sedimentation, Buoyancy Effects, Surface Tension, Surface Energy, Interface Physics, Solid/Liquid Interface, Inclusion and/or Rejection of Particles, Solidification Front Physics, Superconductivity, Processing Difficulties

Number of Samples: It appears that two samples may have been processed in flight.
Sample Materials: aluminum (matrix) with lead droplets (dispersion phase)
(Al*Pb*)
Container Materials: unknown

Experiment/Material Applications:
"There are many engineering applications in which improved performance can be obtained from a metallurgical structure consisting of a uniform dispersion of fine particles of one phase within the bulk matrix phase. The particles may be harder than the matrix to give strengthening, as in the case of dispersion hardened metals, or softer to provide grit embedability[sic], as in the case of plain bearing alloys." (1, p. 89) More specifically, "There is considerable commercial interest in Al-Pb alloys. Early work... was directed towards improving the machinability of aluminum alloys. Subsequently, the bulk of the effort has been directed towards obtaining an improved plain bearing material as a replacement for the intrinsically more expensive aluminum-tin alloys.... More recently, there are claims... that greatly improved superconductivity properties could be achieved from Al-Pb alloys if the structure is carefully controlled." (1, p. 91)

The Al-Pb system was chosen for several reasons. As one example it was noted that "The wide difference between the densities of aluminum (2.3) and lead (10.3) causes rapid Stokes migration [(settling of the dense lead in the aluminum)] in the Earth processed material. The differences between this and the microgravity processed material should therefore be more marked than for alloy systems which have smaller differences between the densities of the two liquids...." (1, p. 91)

References/Applicable Publications:

(1) Caton, P. D. and Hopkins, W. G.: The Preparation and Stability of Metallic Emulsions in Microgravity Environment: An Experiment for the First Space Shuttle Payload (FSLP). In Proc. of the 3rd European Symposium on Material Science in Space, Grenoble, April 24-27, 1979, ESA SP-142, pp. 89-94. (preflight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. Teledyne Brown Engineering Publication (acceleration measurements)

(3) Input received from Experiment Investigator, November 1989.

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Principal Investigator(s): Bethin, J. (1)
Co-Investigator(s): Unknown
Affiliation(s): (1) Corporate Research Center, Grumman Aerospace Corporation, Bethpage, New York

Experiment Origin: USA
Mission: SPAR 10
Launch Date/Expt. Date: June 1983
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Automated Directional Solidification System (ADSS)/Automated Directional Solidification Furnace (ADSF-1, Low Temperature Version) The ADSS was designed to insure that furnace translations resulted in a total ADSS momentum of zero.
Builder of Processing Facility: General Electric Company, King of Prussia, Pennsylvania

Experiment:
Directional Solidification of Magnetic Composites

This SPAR 10 experiment was the third in a series of investigations designed by Bethin and/or Pirich et al. to study the low-gravity directional solidification of a Bi/MnBi eutectic (see Pirich, SPAR 6, SPAR 9 (this chapter)).

During the rocket flight, two hypoeutectic and two hypereutectic samples were solidified in a Bridgman-Stockbarger configuration in the Automated Directional Solidification System (ADSS). A planar solidification interface was produced at approximately 265 ^{o}C. Reportedly, while a furnace gradient of 100 ^{o}C/cm was employed on both SPAR 6 and 9, a 140 ^{o}C/cm gradient was employed on SPAR 10. Further, a furnace velocity of 11 cm/h was chosen in contrast to SPAR 6 (30 cm/h) and SPAR 9 (50 cm/h).

Flight samples were compared to similarly processed samples prepared on the ground. Reportedly, "Macrosegregation... was consistent with a metastable increase in Mn solubility in the Bi matrix, in partial agreement with previous Bi/MnBi SPAR findings of MnBi volume reduction. Smaller mean rod diameter and interrod spacing were found in solidification in low gravity, as compared to Earth gravity, in agreement with previous SPAR findings. In addition, in normal gravity, Mn macrosegregation results for the hypereutectic samples suggest that the thermal instability led to greater convection than did the induced solutal instability. Convection in Earth gravity is suggested as an explanation of morphological differences between normal- and low gravity solidification. This explanation is consistent with a possible change in the equilibrium solubility limit of Mn in Bi observed in low gravity." (1, p. 14)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Eutectics, Magnetic Composites, Magnetic Properties, Hypereutectics, Hypoeutectics, Metallic Matrix, Phase Separation, Binary Systems, Melt and Solidification, Directional Solidification, Bridgman Technique, Thermal Gradient, Solutal Gradients, Thermosolutal Convection, Buoyancy-Driven Convection, Undercooling, Dispersion, Particle Dispersion, Macrosegregation, Solidification Rate, Furnace Translation, Translation Rate, Planar Solidification Interface, Liquid/Liquid Interface, Solid/Liquid Interface, Interrod Spacing, Sample Microstructure, Rod Structure

Number of Samples: four
Sample Materials: bismuth/bismuth-manganese samples of 0.90 and 0.49 wt.% Mn
(Mn*Bi*)
Container Materials: quartz
(Si*O*)

Experiment/Material Applications:
See Pirich, SPAR 6.

References/Applicable Publications:

(1) Bethin, J.: SPAR X Technical Report for Experiment 76-22, Directional Solidification of Magnetic Composites. In Space Processing Applications Rocket (SPAR) Project, SPAR X Final Report, NASA TM-86548, pp. 13-46, July 1986. (post-flight Report)

(2) Bethin, J.: SPAR 10 Technical Report for Experiment 76-22. Directional Solidification of Magnetic Composites, NASA CR-171271, 1984, 51 pp.

(3) Bethin, J.: SPAR X Technical Report for Experiment 76-22, Directional Solidification of Magnetic Composites. Report RE-691, November 1984, 45 pp. (post-flight)

(4) General Electric Company, Space Sciences Laboratory: Operating Manual for Automated Directional Solidification System. Prepared for NASA under Contract NAS8-31536, 1978. (processing facility)

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Co-Investigator(s): None
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Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: College Student Experiment; NASA Get Away Special (GAS) Canister G-033
Volume of Canister 5.0 cubic feet
Location of Canister: STS Payload Bay
<Note: The reflight of this experiment will be on Get Away Special (GAS) Canister G-056.)
Primary Developer/Sponsor of G-033: Caltech Student Space Organization, California Institute of Technology, Pasadena, California/Steven Spielberg
Processing Facility: Container(s) of oil and water mixed by a stirring system and then photographed
Builder of Processing Facility: Unknown

Experiment:
Separation of Oil and Water

This experiment was one of two investigations housed within the G-033 Getaway Special Canister during STS-007. The other experiment within the canister (plant gravireception) was not applicable to this data base. The objective of the de-mixing experiment was to investigate the separation of oil and water in the low-gravity environment.

Specific objectives of the investigation were not detailed in documents which described the STS-007 experiment. However, Reference (6) (listed below) detailed a future Caltech oil-emulsion GAS canister experiment which was to be a reflight of this STS-007 experiment. It is unclear if this future version of the experiment is essentially the same as the experiment flown on STS-007. Reportedly, "The [future] oil emulsion experiment will study the mechanisms of droplet coalescence in various mixtures of oil and water. By using this transparent system instead of liquid immiscible metals, it is possible to photographically record the rate of coalescence between normally immiscible materials that exhibit miscibility in space. The more than 400 exposures taken (in 60 hours as oil and water separate after an initial mixing) should determine feasibility of space-based immiscible alloys production." (6, p. 11)

Two documents published prior to the launch of STS-007 (References (1) and (2)) briefly described the expected STS-007 experimental setup. Initially, the oil and water systems were to be mixed by a motor-driven stirring system. Then the subsequent de-mixing of the components was to be photographed during the following 96 hours. (It appears that the temperature of the fluid was to be controlled throughout the duration of the experiment.) Available references did not further describe the STS-007 experimental setup.

Reportedly, no data were collected from either experiment in the canister (the mixing experiment or plant gravireception) because "A 3-amp fuse... replaced inadvertently with a 1-amp fuse during final safety checks..." (7, p. 26) blew at the moment of payload activation.

No further details which specifically described this STS-007 experiment could be located.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Fluids, Binary Systems, Transparent Liquids, Stirring of Components, Emulsion, Phase Separation, Stability of Dispersions, Liquid/Liquid Dispersions, Droplet Dispersion, Liquid Mixing, Liquid Demixing, Drop Coalescence, Separation of Components, Liquid/Liquid Interface, Contained Fluids, Sample Not Processed As Planned, Fuse Blowout

Number of Samples: unclear
Sample Materials: oil, water
Container Materials: STS-007: unknown; future mission: Lexan™

Experiment/Material Applications:
It was expected that the results of this experiment would "...allow predictions to be made about the possibilities of manufacturing materials such as improved metal alloys and semiconductors in zero-gravity." (2, p. 56)

References/Applicable Publications:
(1) STS-7 Cargo Systems Manual: Gas. JSC-17645 Annex 7 Basic Version PCN-1, NASA JSC, April 1, 1983. (preflight)

(2) NASA STS-7 Seventh Space Shuttle Mission, Press Kit, June, 1983, p. 56. (preflight)

(3) STS-7 Getaway Specials. NASA News, NASA GSFC, May 1983.

(4) Veronda, W.: Space Shuttle: Lab Site for Student Research. Caltech News, June 1983.

(5) Blown Fuse Aborts SSO Experiments Aboard Space Shuttle. Caltech News, October 1983.

(6) Wahl, T. and Barbiere, R. C.: GAS Experiment at Cal Tech. AIAA Student Journal, Fall 1988, pp. 10, 11, and 48. (discusses future GAS flight)

(7) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronauts Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(8) Input received from student familiar with experimental payload, October 1989.

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Experiment Origin: Japan
Mission: TT-500A 13 (Materials Processing Flight #6)
Launch Date/Expt. Date: August 1983
Launched From: Takesaki Launch Site in Tanegashima Island (Tanegashima Space Center, NASDA, Japan)
Payload Type: Sounding Rocket Experiment
Processing Facility: Electric furnace with acoustic mixing
Builder of Processing Facility: Ishikawajima-Harima Heavy Industries Co., Ltd. (IHI), Tokyo, Japan

Experiment:
<u>Al-In: Acoustic Mixing in an Electric Furnace/Graphite Crucible</u>

<No document, published in English, could be located which discussed the objectives, experimental setup, or results of this experiment. The following summary was based on the Principal Investigator's response, August 1988.>

This TT-500A experiment was designed to study the melting and solidification of an immiscible alloy in a furnace equipped with an acoustic mixing device. The objectives of the investigation were to (1) use acoustic mixing to obtain a uniform alloy melt, (2) produce a solidified, uniform immiscible alloy, (3) establish melting and solidification techniques in space, (4) analyze the resulting segregation and agglomeration phenomena in a two liquid system during solidification, and (5) investigate the wetting phenomena between molten metals and crucible materials.

Prior to the rocket flight, an Al-30 mass% In sample (10 mm diameter, 47 mm long) was configured in a graphite crucible. During the low-gravity portion of the mission (reportedly 10^{-4} g), the hyper-monotectic alloy was melted, subjected to acoustic mixing (15 kW, 50 kHz) for 210 seconds, cooled, and solidified.

Post-flight examination of the solidified material revealed that the dispersion of In particles was relatively homogeneous. However, remarkably normal segregation of In was observed in the sample. The In-rich liquid, which separated in the immiscible temperature range, preferentially wetted the crucible wall. A thin film (150 to 200 microns thick) coated the sample surface.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Metallic Matrix, Monotectic Compositions, Hypermonotectic Compositions, Melt and Solidification, Acoustic Mixing, Dispersion, Phase Separation, Homogeneous Dispersion, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Particle Agglomeration, Segregation, Material Interaction with Containment Facility, Wetting, Wetting of Container, Solid/Liquid Interface, Thin Films, Coated Surfaces

Number of Samples: one
Sample Materials: Al-30 mass% In
(Al*In*)
Container Materials: graphite
(C*)

Experiment/Material Applications:
No discussion of the material application could be located in the published literature.

References/Applicable Publications:
(1) Takahashi, T., Kamio, A., Tezuka, H., and Kumai, S.: Solidification of an Immiscible Al-In Alloy. Rep. of NASDA-PSPC-2769, SS58-105 (1983), p. 13.

(2) Takahashi, T., Kamio, A., Tezuka, H., and Kuami, S.: Solidification of Monotectic Alloys in Space. Journal of Japan Institute of Light Metals, Vol. 34, No. 8 (1984), pp. 479-492. (in Japanese)

(3) Input received from Principal Investigator A. Kamio, August 1988.

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab facility, Material Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility (IHF): furnace filled with helium (pressure: 1 bar at 850 °C).
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Solidification of Immiscible Alloys Part 1- Binary Systems: Zn-Bi, Zn-Pb (1ES301) and Part 2- Ternary System: Zn-Bi-Pb (1ES306)

During both ground-based and space-based investigations, homogeneous melts of monotectic alloys have separated into two phases during cooling. It was, therefore, concluded that gravity is not the primary driving force for this separation. Studies indicated that the separation was greatly dependent on either (1) the volume content of the minority phase or (2) the interfacial energy differences between sample components.

This Spacelab 1 experiment was the third in a series of investigations designed by Ahlborn and/or Löhberg et al. to study the solidification of metallic alloys (see Löhberg, SPAR 2 (Chapter 17) and Ahlborn, TEXUS 1 (Chapter 6)). The specific objective of the experiment was to determine the cause of of the phase separation of immiscible systems by "...using specimens from two different binary systems and from the ternary system formed from these two binary systems, to... [obtain a] continuous transition from the interfacial energy of one to the other system." (1, p. 55)

During the mission, 7 samples of Zn-Bi, 7 samples of Zn-Pb, and 14 samples of Zn-Bi-Pb were heated to 850 °C in the Spacelab Isothermal Heating Facility. <Note: The specific compositions of these 28 samples were reported in figures 1 and 2 of Reference (1). Compositions ranging from a few volume % Zn to nearly 100 volume % Zn were selected. (See Reference (1) for more details.)> After a soak time of 15 minutes, the samples were cooled at a rate of nearly 30 K/min through the miscibility gap. It was noted that both axial and radial temperature gradients ex-

isted within the crucibles.

<Note: Processing of the 7 Zn-Bi samples and the 7 Zn-Pb samples was achieved during the first experiment (designated as 1ES301); processing of the 14 Zn-Bi-Pb samples was achieved during the second experiment (1ES306). The two experiments were processed one after another and had nearly identical time-temperature profiles. Although it is unclear, it appears that a single cartridge held 14 samples during each processing run.>

Post-flight examination of the samples indicated that an inhomogeneous distribution of the minority phase occurred and that there was essentially a pronounced enrichment of minority phase droplets in the part of the specimen which experienced the hottest temperature. "Obviously the droplets formed during cooling through the miscibility gap were transported to this hotter side." (1, p. 57) Droplet size differences between specimens were noted, and generally the Zn-rich alloys had much smaller drops than the Pb or Bi rich specimens. While it was concluded that the size of the droplets was "...mainly determined by the volume portion of the minority phase, the temperature interval... [had] no valuable influence." (1, p. 55)

"The results show that the separation process in monotectic alloys is governed by the growth of the precipitated droplets of the minority phase and their transport to the hotter part." (1, p. 59) Two droplet growth processes dependent on the interface energies and viscosities of the employed melts were discussed in light of the results: (1) coagulation by collision and movement of droplets by a Marangoni force and (2) coagulation migration due to the overlapping of diluted zones around the droplets (see Reference (1)).

It was concluded that the results of the experiment did not indicate which separation process(es) were the most important during the experiments. Reportedly, if a homogeneous distribution of small sized droplets of the minority phase in a liquid matrix is desired, future experiments must have a minority volume portion no greater than 5%, "...a low interfacial energy between the droplets and the matrix and a high viscosity of the matrix." (1, p. 60)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Metals, Monotectic Compositions, Binary Systems, Ternary Systems, Metallic Matrix, Melt and Solidification, Drops, Coagulation, Droplet Collision, Drop Migration, Droplet Size, Particle Growth, Particle Motion, Metallic Dispersion, Homogeneous Dispersion, Liquid/Liquid Dispersion, Minority Phase, Separation of Components, Marangoni Force, Marangoni Movement of Droplets, Interfacial Energy, Viscosity, Density Difference, Precipitation of Second Phase, Phase Separation, Separation of Components, Liquid/Liquid Interface, Solid/Liquid Interface, Thermal Soak, Thermal Gradient, Solidification Rate

Number of Samples: twenty-eight
Sample Materials: Zn-Bi, Zn-Pb (experiment 1ES301) and Zn-Bi-Pb (experiment 1ES306)
(Zn*Bi*, Zn*Pb*, Zn*Bi*Pb*)
Container Materials: graphite
(C*)

Experiment/Material Applications:
This experiment sought to investigate separation processes which occur in both space and ground-based processing of homogeneous, monotectic alloys cooled through the miscibility gap. Differences in density of the molten phases causes separation to occur, and resultant alloys may not be fit for some technical applications.

The Zn-Pb, Zn-Bi and Zn-Pb-Bi systems were chosen for many reasons including the following:

"-one component (Zn) is common for all systems, providing the same matrix for precipitation of different droplets.

"-the monotectic temperatures of the alloys from the binary and ternary systems are nearly the same (418-416 ^{o}C)....

"-the temperature intervals in which precipitation of droplets occurs can be varied widely for the same volume portion of the minority phase...." (1, p. 56)

(Additional reasons are reported in Reference (1), p. 56)

References/Applicable Publications:

(1) Ahlborn, H. and Löhberg, K.: Influences Affecting Separation in Monotectic Alloys Under Microgravity. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 55-61.

(2) Chassay, R. P and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements on Spacelab)

(3) Whittman, K: The Isothermal Heating Facility. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA Publication ESA SP-222, pp. 49-54. (IHF facility)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab facility, Materials Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility (IHF) Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Separation of Immiscible Melts (W1-IHF-01)

This Spacelab D1 experiment was the fourth in a series of investigations designed by Ahlborn and/or Löhberg et al. to study the solidification of metallic alloys (see Löhberg, SPAR 2 (Chapter 17), Ahlborn, TEXUS 1, Spacelab 1 (Chapter 6)). Earlier research in this investigative series (designed to investigate the processes governing separation of immiscible alloys in both ground-based and space-based laboratories), was extended on this Spacelab D1 mission. The specific objectives of this experiment were (1) to study the transport of minority-phase droplets due to the Marangoni force and (2) to find a composition at which a homogeneous distribution of these droplets would result.

Eleven binary (Zn-Pb, Zn-Bi) and ternary (Zn-Pb-Bi) systems (similar to those examined during Spacelab 1) were employed. In addition, three Al-Pb systems were processed. <Note: The volume percentages of each of the materials was not specifically stated, although they can be somewhat derived from Reference (5).> Two of the Al-Pb specimens "...consisted of a pure Al-ingot in which Pb balls with 2 and 4 mm diameter were incorporated in the molten state. The third specimen consisted of pure Pb in which an Al cylinder (4mm long, 4mm diameter) has been mechanically embedded. At the temperature of 850 deg. C planned for this experiment, these specimens are still within the miscibility gap. Accordingly, the Marangoni transport to the hotter side should take place during holding at this temperature." (5, p. 298)

Samples were to be processed similarly to samples solidified during Spacelab 1 with a radial temperature gradient available to initiate and sustain Marangoni droplet transport. Unfortunately, a more pronounced axial temperature gradient existed in the D-1

experiment than planned.

Post-flight analysis of the samples confirmed Spacelab 1 observations. "The size of the minority phase droplets in the same system depends mainly on its volume content. Although there existed... [a] stronger axial temperature gradient inside the specimens, the influence of the radial gradient on the transport of the minority phase droplets towards the inner (hotter) side was confirmed too." (5, p. 301) "The Pb droplets incorporated in the Al-specimens were largely dissolved due to a higher temperature in the furnace than planned. A Marangoni-transport of the larger droplets to the side of the specimen with the higher temperature gradient could not be observed." (1, p. 80)

<Note: Further discussions of the separation mechanisms related to differences in viscosities of the melt, size and distribution of droplets of the minority phase, etc. were presented in Reference (5). A small section of this discussion as it related to the experimental objectives, is reproduced below:>

"The size and the distribution of droplets of the minority phase in the Zn-rich matrices show that it will be very difficult to get homogeneous distributed droplets with diameters smaller than 1 μm, even if the gravity driven segregation is avoided.... [T]he temperature gradient gives rise to the Marangoni motion even in case of equal volume fractions of both components. The Marangoni motion will be slow because of the small size of the droplets of the minority phase in the Zn-Pb or Bi based matrix. Interface energies are equal in both cases if the temperature interval of the precipitation does not differ. Accordingly, it seems that the viscosities of the fluid phases play a dominant role. The lower the viscosity of the liquid matrix, the higher is the velocity of the Marangoni motion and the more probable the collision of droplets, the larger will be their final size." (5, p. 302)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Metals, Binary Systems, Ternary Systems, Metallic Matrix, Melt and Solidification, Drops, Droplet Collision, Drop Migration, Droplet Size, Particle Size Distribution, Particle Motion, Droplet Dispersion, Metallic Dispersion, Homogeneous Dispersion, Liquid/Liquid Dispersion, Minority Phase, Separation of Components, Marangoni Force, Marangoni Movement of Droplets, Marangoni Movement of Droplets, Interfacial Energy, Viscosity, Composition Distribution, Density Difference, Segregation, Precipitation of Second Phase, Phase Separation, Separation of

Components, Liquid/Liquid Interface, Solid/Liquid Interface, Thermal Gradient, Thermal Environment More Extreme Than Predicted, Sample Evaporation

Number of Samples: fourteen
Sample Materials: Zn-Bi, Zn-Pb, Al-Pb, and Zn-Pb-Bi. <Note: See Reference (5) for more information.>
(Zn*Bi*, Zn*Pb*, Zn*Pb*Bi*, Al*Pb*)
Container Materials: Unknown, possibly graphite. (Spacelab 1 experiments employed graphite crucibles.)
(C*)

Experiment/Material Applications:
See Ahlborn, Spacelab 1.

References/Applicable Publications:
(1) Ahlborn, H. and Löhberg, K.: Separation of Immiscible Alloys Under Reduced Gravity. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1 Symposium, Norderney, Germany, August 27-29, 1986, p. 80. (abstract only)

(2) Ahlborn, H. and Löhberg, K.: Separation nicht mischbarer Schmeltzen unter verminderter Schwerkraft. In Naturwissenschaften, 73.Jahrgang Heft 7, July 1986, pp. 378-380. (in German)

(3) Ahlborn, H.: Separation of Immiscible Melts. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 131-132. (preflight)

(4) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 48-58. (post-flight; acceleration measurements)

(5) Ahlborn, H. and Löhberg, K.: Separation of Immiscible Alloys Under Reduced Gravity. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 297-304. (post-flight)

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Experiment Origin: Austria
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Material Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility (IHF) furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Solidification of Near Monotectic ZnPb Alloys (1ES313)

This Spacelab 1 experiment was the first in a series of investigations designed by Fischmeister et al. to study the solidification of immiscible alloys under low-gravity conditions. The specific objectives of this experiment were to (1) determine if Zn-Pb alloys could be maintained in the dispersed state within the miscibility gap and (2) examine the coarsening behavior of the lead particles within the system.

The sample preparation was different from previous low-gravity experiments (e.g., see Fredriksson, TEXUS 2; Fredriksson, TEXUS 14b (this chapter)): (1) the samples contained a low volume fraction (below 2 vol.%) of the dispersed droplets (Pb) and (2) the samples contained a finely dispersed two-phase structure (obtained by quench casting). The low volume fraction samples reduced the probability of demixing by coalescence of the particles; the initial finely dispersed structure resulted in a retention of the Pb-rich particles as droplets which excluded the nucleation of droplets from a single phase melt.

Prior to the mission, two sets of samples of the Zn-Pb alloy (2 wt.%, 2.5 wt.%, 3 wt.%, 4 wt.%, and 5 wt.%) were prepared by quench casting in either Cu or cast iron molds. The different mold materials were used to produce different Pb particle sizes (sizes ranged from 1.1 to 9 microns; see Reference (1) for details). The specimens were machined to 5.0 mm diameter and 15 mm length. "Three of these were enclosed in a tantalum tube of 0.5 mm wall thickness to contain the Zn vapour pressure in the event of gross overheating of the... [furnace].... Six ampoules were jointly placed in a graphite block in an IHF [Isothermal

Heating Facility] cartridge, with three thermocouples monitoring the temperature at opposite ends and in the center of three symmetrically placed ampoules." (1, p. 64) <Note: This description of the specimen configuration was not clear to the editors. Information provided by the Principal Investigator (Reference (7)) seems to imply the following: (1) the above quote refers to 18 specimens, (2) the 18 specimens were placed in 6 tantalum crucibles (3 specimens per crucible), and the 6 crucibles (ampoules) were placed in 1 IHF cartridge.>

During the Spacelab 1 mission, the Isothermal Heating Facility (IHF) was used to melt and resolidify the samples. The samples were heated to 475 ^{o}C and held at that temperature for 60 minutes. Reportedly, "The temperature of the isothermal hold had been intended to be 450 ^{o}C, which would have left all samples with 2 wt.% Pb in dispersion. Overheating in the IHF to 475 ^{o}C homogenized this alloy, and on cooling L_2-droplets had to nucleate." (4, p. 162)

"Two microgravity runs were planned, each with a set of specimens with different volume fractions and initial particle sizes, to secure mutually supporting data on particle coarsening. [The second run was to be performed at a higher temperature in order to study the temperature influence on Ostwald ripening.] Unfortunately, the second run... [had] to be cancelled after a system malfunction which severely curtailed available IHF time." (1, p. 64) <Note: The number of samples processed during each run was not clear. It appears that each set consisted of 18 specimens.> Further, while the sample crucibles were designed with a volume expansion mechanism, "Because of an error in manufacturing, the specimens were instead enclosed with a tight fit in the tantalum ampoules. This led to fracture and expulsion of some of the melt in several flight samples." (1, p. 64) This problem may have resulted in a higher level of convection in the sample melt than originally planned.

It was reported that "about half of the 18 samples were affected" by the fracture and expulsion of the melt by volume expansion. "...six were strongly affected and 3... [slightly affected] by partial fracture of the tantalum ampoules, so that 9 samples could be used for careful examination." (Reference (7))

Postflight analysis of the flight and 1-g processed reference samples included light metallography and scanning electron microscopy examinations. Data were reported in terms of "diameters of circles of area equivalent to particle intersects." No attempt was made to determine three-dimensional particle diameters because of the accumulation of error in the numerical integration procedures. It was reported that very little migration of the Pb particles toward specimen edge occurred. The

samples largely exhibited a homogeneous structure. However, the particles in all samples did undergo a significant amount of coarsening. Plotting the amount of coarsening versus time (diffusion-controlled coarsening according to the LSW model, see Reference (1)) indicated a strong dependence on initial volume fraction of the Pb-rich droplets. <Note: 'LSW' refers to the theory of Ostwald Ripening by Lifshitz, Slyozov, and Wagner.> Analysis led to the conclusion that coarsening was attributed to an Ostwald mechanism rather than coalescence due to convection mechanisms. "In view of the lack of reliable diffusivity data, we can only conclude that the identification of Ostwald ripening as the main mechanism of particle coarsening... is not contradicted by available data." (1, p. 68)

<Note: Reference (5) was not translated prior to the preparation of this experiment summary. Thus, the information within Reference (5) is not represented in this summary.>

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Monotectic Compositions, Metallic Matrix, Binary Systems, Melt and Solidification, Drops, Phase Separation, Metallic Dispersion, Homogeneous Dispersion, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Particle Dispersion, Stability of Dispersions, Ostwald Ripening, Particle Coarsening, Particle Coalescence, Particle Size Distribution, Drop Migration, Particle Migration, Nucleation, Separation of Components, Liquid Demixing, Diffusive Mass Transfer, Thermal Soak, Isothermal Processing, Solid/Liquid Interface, Volume Compensation, Volume Expansion, Liquid Leakage, Liquid Transfer, Thermal Environment More Extreme Than Predicted, Processing Difficulties, Hardware Malfunction, Sample Not Processed As Planned

Number of Samples: eighteen
Sample Materials: Zn-Pb samples with 2 wt.% Pb, 2.5 wt.% Pb, 3 wt.% Pb, 4 wt.% Pb, or 5 wt.% Pb
(Zn*Pb*)
Container Materials: tantalum crucibles contained within graphite blocks
(Ta*, C*)

Experiment/Material Applications:
No discussion of the material application could be located in the published literature.

References/Applicable Publications:

(1) Kneissl, A. and Fischmeister, H.: Particle Coarsening in Immiscible Zinc-Lead Alloys Under Microgravity. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 63-68. (post-flight report)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements)

(3) Whittmann, K.: The Isothermal Heating Facility. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA Publication ESA SP-222, pp. 49-54.

(4) Ratke, L., Fischmeister, H., and Kneissl, A.: Coarsening of Liquid Zn-Pb Dispersions - A Spacelab Experiment. In Proceedings of the 6th European Symposium on Material Sciences under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 161-167. (post-flight)

(5) Kneissl, A. and Fischmeister, H. F.: Schmelzen und Erstarren von übermonotektischen Zink-Blei-Legierungen unter Schwerelosigkeit. Metall 38 (1984), pp. 831-837. (in German)

(6) Coarsening of Liquid Zn-Pb Dispersions- Final Evaluation of a Spacelab 1 Experiment. Proceedings VII European Symposium on Materials and Fluid Sciences in Microgravity, Oxford, UK, September 10-15, 1989, ESA SP-295 (January 1990), pp. 135-140. (postflight)

(7) Input received from Principal Investigator A. Kneissl, August 1993.

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility (IHF) furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Ostwald Ripening in Metallic Melts-Interfacial Phenomena and Transport Processes in Binary Monotectic Alloys (WL-IHF 04)

This Spacelab D1 experiment was the second in a series of investigations designed by Fischmeister et al. to study the solidification of immiscible alloys under low-gravity conditions (see Fischmeister, Spacelab 1). The specific objectives of the experiment were to (1) study Ostwald Ripening in Al-In alloys of different compositions, (2) examine the development of grain boundary grooves at the interface between Al-bicrystals and an Al-In melt, and (3) study "...a non-equilibrium reaction at the contact face between solid Al and an In-rich Al-In alloy producing a layer of another Al-rich equilibrium phase whose thickness gives information about diffusion coefficients." (4, p. 339)

During the Spacelab D1 mission, several Al-In immiscible alloy samples of different compositions were processed using the Isothermal Heating Facility. However, the experiment "...failed almost completely due to a malfunction of the thermocouples such that the samples were overheated above a critical temperature. It was therefore impossible to study Ostwald ripening and compare it with the theories worked out during the preparation of this experiment for the D1-mission. One sample show[ed] strong coarsening, probably due to collisions and coagulation, whereas all other[s] exhibit massive separation into two liquids. Although our single D1 experiment on Ostwald ripening failed, the experiment as a whole (including ground based theoretical and experimental research in the pre- and post-mission phase) was a step forward to a better understanding of separation processes of immiscible liquid alloys." (1, p. 43)

(Reference (4) contains a discussion of the theoretical work for this experiment.)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Monotectic Compositions, Metallic Matrix, Binary Systems, Melt and Solidification, Drops, Phase Separation, Metallic Dispersion, Homogeneous Dispersion, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Particle Dispersion, Stability of Dispersions, Ostwald Ripening, Particle Coarsening, Coagulation, Droplet Collision, Drop Migration, Particle Migration, Nucleation, Separation of Components, Liquid Demixing, Diffusive Mass Transfer, Diffusion Coefficient, Solid/Liquid Interface, Interface Physics, Interface Phenomena, Grain Boundaries, Thermal Environment More Extreme Than Predicted, Hardware Malfunction, Processing Difficulties

Number of Samples: unknown, possibly four
Sample Materials: Al-In alloys of various compositions
(Al*In*)
Container Materials: graphite within a tantalum sheath
(Ta*, C*)

Experiment/Material Applications:
No discussion of the material application could be located in the published literature.

References/Applicable Publications:
(1) Ratke, L., Thieringer, W. K., and Fischmeister, H.: Coarsening of Immiscible Liquid Alloys by Ostwald Ripening. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, pp. 42-44. (post-flight results; abstract only)

(2) Fischmeister, H. F., Ratke, L., and Thieringer, W.: Ostwald Ripenning in Metallic Melts. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 145-146. (preflight)

(3) Whittmann, K.: The Isothermal Heating Facility. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 49-54. (published post-Spacelab 1 but prior to D1)

(4) Ratke, L., Thieringer, W. K., and Fischmeister, H.: Coarsening of Immiscible Liquid Alloys by Ostwald Ripening. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 332-341 (specifically pp. 339-340). (post-flight)

(5) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986. (acceleration measurements on D1)

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Experiment Origin: Saudi Arabia
Mission: STS Launch #18, STS-025 (STS 51-G, Discovery)
Launch Date/Expt. Date: June 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Phase Separation Experiment (PSE) Hardware (a 5 x 4 x 1-inch hand-held apparatus containing 15 separation chambers)
Builder of Processing Facility: Developed at the National Aeronautics and Space Administration (NASA), Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Phase Separation Experiment (PSE)

The objective of this STS 51-G experiment was to study the low-gravity mixing and subsequent separation of several immiscible-liquid two-phase systems.

Prior to the mission, 15 experiment chambers, configured within a hand-held, transparent Plexiglas™ container, were filled with various two-phase systems. An orange, green, or blue dye was added to at least one fluid in most of the chambers to enhance photographic resolution of the separation. (The contents of each chamber can be found in the Sample Materials section below.) Reportedly, the specific liquid systems were selected to illustrate "...the effects of a number of... variables such as density, interfacial tension, viscosity, and composition..." (4, p. 356) on the separation process.

Chambers 1 through 5 of the experimental apparatus were double walled: the inner wall a glass cuvette of 1.38 ml volume, the outer wall a Plexiglas™ chamber. Chambers 6 through 15 were single-walled Plexiglas™ chambers of 3.46 ml volume. Each chamber was equipped with a small metal ball or glass mixing bead(s).

During the mission, the 15-chambered apparatus was removed from storage, mounted in front of a light source, and photographed. (Each photograph showed all 15 chambers.) The apparatus was then removed from the mount and shaken by the Payload Specialist. The steel balls and glass beads in the chambers facilitated the mixing of the two fluid phases. After mixing, the container was

re-mounted in front of the light source and the subsequent demixing of the fluids photographed. During the first 2 minutes (after shaking) one photograph was taken every 15 seconds; during the next 10 minutes, one picture was taken every minute. Two hours later, the undisturbed apparatus was photographed again.

It appears from Reference (4) that the experiment sequence (mounting, shaking, remounting, and filming) was performed four times on the mission with the single 15-chamber apparatus. Color pictures of the apparatus at different times during the experiment can be viewed in Reference (4).

After post-flight examination of the flight photographs it was reported "that the mixing is accompanied by a noticeable emulsification of the chamber's components. The mixing appears, however, to be inadequate in the top row as compared to... [other chambers]. This seems to be due to the fact that glass mixing balls were used and they were not dense enough to mix the phases in low-gravity....

"The phases in chambers 1,4,6,7,9,10,11, and 15 appear to be well mixed.... The phases in chambers 3,8,12, and 13 are moderately mixed and the contents in chambers 2 and 5 are not mixed.... <Note: although not stated at this point but briefly alluded to later, it appears that the contents of chamber 14 also mixed well.> It is obvious from the pictures that... [generally], glass mixing balls proved inadequate to mix the phases having high interfacial tension.... [C]hambers 2 and 8 both contained hexadecane and distilled water but chamber 8... [had] a Plexiglas™ wall and a steel ball rather than a glass wall and a glass ball as in chamber 2. Chamber 8 showed little emulsification and some reforming of two phases whereas Chamber 2 showed some initial coalescence of the hexadecane-water system; but generally they did not mix. This may be due to high interfacial tension and the lack of polar impurities (surfactants) in the hexadecane-water system. This explanation is further supported by the observation of chambers 9 and 10. The contents in these chambers contained a surfactant detergent (chamber 9) and saline water (chamber 10). They emulsified easily and stayed emulsified. This behavior is attributed to the ability of surfactants to lower interfacial tension in these airless liquid-liquid two-phase systems.

"Some air bubbles were observed in a few chambers. Also, in the absence of gravity instead of layer formation, droplet fusion occurred, forming ever growing regions of each phase." (4, pp. 359-360) <Note: It is assumed that the preceding sentence means that rather than the phases separating into layers, a droplet of one phase occurred, with slow diffusion of the other phase into this drop.>

The phase separation behavior of the STS 51-G experiment was compared to separation behavior observed during similar experiments performed on Earth. <Note: Reference (4) did not always specifically state the observed behavior of the terrestrial system, but instead, detailed the results of the flight experiment.> The following was reported (Reference (4)):

<Note: See the **Sample Materials** section for the contents of each chamber.>

Chamber 1: The nickel precipitate stayed mixed in low-gravity and particles did not aggregate.
Chamber 2: the hexadecane-distilled water system showed some initial coalescence, but generally did not mix.
Chambers 3, 4, and 5: The fluorinated Krytox oil in these chambers is denser than the second phase, water. The contents in chambers 3 and 4 mixed slightly, but the contents in chamber 5 did not.
Chambers 6 and 7: On Earth, phases in both chambers separated well. In space, the phase in chamber 6 remained as an emulsion. The phases in chamber 7 formed a fairly stable emulsion after mixing but had separated 2 hours later.
Chamber 8: The results from this chamber were discussed above.
Chamber 9: A detergent additive to the contents of this chamber (hexadecane and distilled water) allowed the study of the effect of detergent on the interfacial tension of a liquid-liquid system. On Earth, the system demonstrated good separation; in space, the system remained emulsified.
Chamber 10: The hexadecane/saline-water solution remained emulsified in space.
Chambers 11 and 12: These chambers contained equal volumes of gas-oil/distilled water as in chamber 7 but different amounts of glass beads (to create difference surface areas). The phases in both chambers separated in space prior to mixing (the beads in oil). When mixed, an emulsion formed, which slowly "cleared."
Chamber 13: This chamber illustrated that the employed hexadecane/water solution is more difficult to emulsify than gas-oil/water.
Chamber 14: A stable emulsion formed in this hexadecane-water system with detergent additive.

<Note: Reference (4) which was provided by the Principal Investigator, was missing page 366, the page which detailed the results of the capillary wetting experiment of chamber 15 as well as other pertinent information. Efforts are being made to secure this missing page.>

It was concluded that in the absence of gravity, "...phase separation depends largely on interfacial tension and mixing efficiency. For example the emulsions of oil-saline water

(chambers 6 and 7) remained mixed for a long time... [in] microgravity, at one gravity the two phases separated easily. Similarly, when the surfactant was used to lower interfacial tension, stable emulsions were formed at microgravity (chamber 9), although at one gravity both hexadecane and distilled water showed good separation." (4, p. 367)

Recommendations concerning future phase separation experiments included the following: (1) using video photography to provide a continuous record of phase behavior coupled with time lapse documentation, (2) improving the experimental apparatus which was "inadequate to obtain clear data, especially when capillary studies... [were] involved." (1, p. 20), and (3) improving the fluid mixing within the experimental apparatus.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Fluids, Binary Systems, Ternary Systems, Transparent Liquids, Liquid Mixing, Stirring of Components, Emulsion, Dispersion, Liquid/Liquid Dispersion, Droplet Dispersion, Particle Growth, Particle Dispersion, Stability of Dispersions, Drops, Drop Coalescence, Particle Aggregation, Liquid Demixing, Separation of Components, Phase Separation, Phase Partitioning, Two-Phase System, Liquid/Liquid Interface, Liquid/Gas Interface, Bubbles, Capillary Forces, Wetting, Surface Tension, Interfacial Tension, Viscosity, Surfactants, Diffusive Mass Transfer, Density Difference, Contained Fluids, Illumination System

Number of Samples: fifteen
Sample Materials: The following materials were reported (Reference (4)):
Chamber 1 (nickel precipitate experiment): one volume each of a water-ethanol system containing 0.55 M alpha furil dioxine, 0.25 M nickel chloride plus 0.5% v/v sodium hydroxide and one large glass mixing ball.
Chamber 2: One volume each of hexadecane-distilled water and one large glass mixing ball.
Chamber 3: One volume each of Krytox oil-distilled water and one large glass mixing ball.
Chamber 4: One volume/3 volumes Krytox oil-distilled water and one large glass mixing ball.
Chamber 5: Three volumes/1 volume Krytox oil-distilled water and one large glass mixing ball.
Chamber 6: One volume each of gas-oil - saline water and one stainless steel mixing ball.
Chamber 7: One volume each of gas-oil - distilled water and one

stainless steel ball.
Chamber 8: One volume each of hexadecane-distilled water and one stainless steel ball.
Chamber 9: One volume each of hexadecane-distilled water containing 0.4% w/w Igebal CO-710 detergent and one stainless steel ball.
Chamber 10: One volume each of hexadecane-saline water and one stainless steel ball.
Chamber 11: 1.23 ml each of gas-oil - distilled water and 2.2 g (1 ml) of medium glass beads.
Chamber 12: As in chamber 11 but small glass beads were used.
Chamber 13: As in chamber 11 but hexadecane used instead of gas-oil.
Chamber 14: As in chamber 13 but water contains Igebal detergent as in chamber 9.
Chamber 15: 0.86 ml. of green dyed water (25% of chamber volume) in a chamber containing (filled) with 28 large glass beads occupying approx. 1.75 ml = 50% of the chamber volume.

"Hexadecane, gas-oil, and Krytox oil were stained with Sudan Orange (0.1 mg/ml). The water in chamber 15 was stained with 1% w/w Methyl Green and in chambers 3,4, and 5 with Trypan Blue (0.1 mg/ml).

Other details of the materials can be found in Reference (1).

Container Materials: Chambers 1-5: glass enclosed in Plexiglas™; chambers 6-15: Plexiglas™

Experiment/Material Applications:
Although it was not stated in the available references, it is suspected that these experiments were initiated to study the possibility of producing improved alloys or semiconductors from immiscible materials.

References/Applicable Publications:
(1) Dabbagh, A. E.: STS 51-G Post Mission Report of Saudi Arabian Payload Specialists and Arabsat Scientific Experiments Team. Arabsat Scientific Experiments Project, July 1985, p. 20. (post-flight)

(2) NASA Space Shuttle Mission 51-G Press Kit, June 1985, p. 15. (preflight)

(3) Input received from Principal Investigator M. Z. El-Faer, June 1991.

(4) El-Faer, M. Z., Ali, M. F., Asar, H. K., and Al-Saud, I. S.: Phase Separation in Microgravity Evaluation of Arabsat Phase Separation Experiment Results. In The Arabian Journal for Science and Engineering, Volume 13, No. 3. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Fluid Physics Module (FPM) (same facility as the FPM of Spacelab 1 but with improvements)
Builder of Processing Facility: FIAT Centro Ricerche, Italy

Experiment:
Mixing and Demixing of Transparent Liquids (Run A and B) - the additional effects of a free surface on mixing and demixing (WL-FPM-03)

This Spacelab D1 experiment was the fifth in a series of investigations designed by Langbein and/or Heide et al. to study the behavior of immiscible systems under low-gravity conditions (see Heide, TEXUS 5, TEXUS 7, TEXUS 8, TEXUS 9 (all in this chapter)). The specific objective of the experiment was to examine the fluid mechanisms operating during the mixing and demixing of binary transparent liquids exhibiting a miscibility gap. Such liquid systems are used as models for metallic alloys whose compounds separate during solidification.

The experiment was performed in a closed liquid container attached to the Spacelab D1 Fluid Physics Module (FPM). The container held two concave, aluminum disks, between which a cylindrical column could be formed. Because the test liquid (benzylbenzoate and paraffin oil) had a low contact angle with aluminum, the disks had sharp edges and were surrounded by teflon rings. The front disk contained a heater redundantly controlled by two thermistors.

During the first part of the experiment procedure, the liquid column was formed by injecting test liquid through a hole in one of the disks and, simultaneously, separating the disks by "...rear plate rotation of the FPM." (4, p. 118) The front disc was then heated and maintained at the desired temperature. The liquid was cooled by passive radiation and conduction.

Although initially only one experimental run had been planned, two experimental runs were performed during the mission.

During Run A, buildup of the liquid column took approximately 10 minutes. The front disk was then heated to approximately 60 ^{o}C. Reportedly, because of "...liquid mixture spread[ing] across the teflon rings surrounding the supporting metallic disks, the column obtained formed an unduloid rather than a cylinder." (9, p. 325) The 60 ^{o}C temperature was maintained for approximately 22 minutes. Passive cooling was then allowed to occur. During the first 46 minutes of the experiment, a TV downlink allowed interaction between the Payload Specialist and ground. For approximately 10 minutes after the TV downlink was halted, a Vinten camera recorded the behavior of the liquid column. The recording was then halted until the liquid was sucked back into the reservoir.

Because of the "successful" performance of the first experimental run, a second experiment (Run B) was performed. The procedure was the same as Run A except that (1) the heater temperature was increased to 75 ^{o}C (to increase the Marangoni velocity and distinguish between reproducible and irreproducible phenomena) and (2) "...only the last seconds of the heating phase, the cooling phase and the recovery of the column [were] recorded." (9, p. 326)

The conclusions from both experimental runs were reported as follows:

(1) The mechanisms of capillarity, stability, and spreading are significant during the mixing and demixing of fluids exhibiting a miscibility gap. These mechanisms control the final distribution of the two components.

(2) Marangoni convection caused by non-uniform heating and cooling was lower than expected. The convection differed by at least one order of magnitude from that determined by ground experiments. This difference was attributed to (a) contamination of the liquid mixture from long periods of storage and/or (b) "...contrasting effects of temperature and concentration on the surface tension due to the component having the lower surface tension (paraffin oil) getting enriched on the cold side." (9, p. 327) If the difference in Marangoni convection can be ascribed to (b), "...the advantage of a free fluid surface, the suppression of heterogeneous nucleation, will not generally be balanced by the disadvantage of stronger Marangoni convection. In that case containerless processing appears commendable also for metallic alloys." (9, p. 327) <Note: This point, as written, was not clear to the editors.>

(3) Slow cooling reduced the demixing of the two liquids, suggesting this result may also be true for metallic alloys.

(4) The diffuse interface layer between the two liquids that mixed exhibited properties of normal interfaces (e.g., capillarity, stability, spreading effects and Marangoni convection).

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Fluids, Binary Systems, Phase Separation, Transparent Liquids, Model Materials, Liquid Columns, Liquid Bridges, Liquid Bridge Stability, Liquid Stability, Liquid/Liquid Interface, Liquid/Liquid Dispersion, Liquid Mixing, Liquid Demixing, Separation of Components, Free Surface, Surface Tension, Contact Angle, Liquid Spreading, Capillary Forces, Meniscus Shape, Free Surface Shape, Thermocapillary Convection, Marangoni Convection, Thermosolutal Convection, Solutal Gradients, Nucleation, Heterogeneous Nucleation, Thermal Gradient, Wetting, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Passive Cooling, Radiative Cooling, Conduction, Cooling Rate, Contamination Source, Deterioration of Loaded Samples Prior to Launch, Containerless Processing Applications

Number of Samples: two experiment runs
Sample Materials: Nontoxic paraffin oil/benzylbenzoate (interface tensions and contact angles are given in the available publications); critical temperature: 61 ^{o}C; flammability temperature: 93 ^{o}C.
Container Materials: Not applicable. Free surface liquid bridge was formed between two aluminum disks.
(Al*)

Experiment/Material Applications:
The liquid system (cyclohexane/methanol) used during the TEXUS experiments (see Heide, TEXUS 7; Heide, TEXUS 9) could not be employed for this experiment because of safety precautions (toxicity, flammability). Therefore, the model system benzylbenzoate/paraffin oil was used.

References/Applicable Publications:

(1) Langbein, D. and Heide, W.: Study of Convective Mechanisms Under Microgravity Conditions. Advances in Space Research, Vol. 6, Number 5, 1986, pp. 5-17. (post-flight; discusses results from TEXUS 7, TEXUS 9 and Spacelab D1)

(2) Langbein, D. and Heide, W.: Mischen und Entmischen transparenter Flussigkeiten. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1- Symposium, Norderney, Germany, August 27-29, 1986, pp. 81-84. (in German)

(3) Langbein, D.: Mixing and Demixing of Transparent Liquids. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 139-140. (preflight)

(4) Langbein, D. and Heide, W.: Mixing and Demixing of Transparent Liquids Under Microgravity. In 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 117-123.

(5) Gonfalone, A.: The Fluid Physics Module- A Technical Description. In ESA 5th European Symposium on Materials Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 3-7. (status post-Spacelab 1, prior to D1)

(6) Ceronetti, G.: The Fluid Physics Module Design. XXV Rassegna Internazionale Elettronica Nucleare Ed Aerospaziale. Roma, March 10-19, 1978, pp. 76-83. (prior to Spacelab 1)

(7) Martinez, I., Haynes, J. M., and Langbein, D.: Fluid Statics and Capillarity. In Fluid Sciences and Materials Science in Space, Edited by H. U. Walter, Springer Verlag, 1987, pp. 53-80. (related topics)

(8) Langbein, D.: Fluid Physics. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986. (specifically pp. 101-102; post-flight)

(9) Langbein, D. and Heide, W.: Mixing and Separation in Transparent Liquids Under Microgravity. In Proceedings of the Norderney Symposium in Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 321-327. (post-flight)

(10) Input received from Principal Investigator D. Langbein, August 1993.

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Co-Investigator(s): Sprenger, S. (3)
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 13
Launch Date/Expt. Date: April 1986
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Isothermal Heating Furnace
The experiment module was equipped with a total of four furnaces, two of which were used for this study. <Note: While the Principal Investigator reported that the module designation was TEM 01-2, Reference (6) indicates that the module designation was TEM 01-1.>
Builder of Processing Facility: ERNO Raumfahrttechnik GmbH, Germany

Experiment:
Separation in Monotectics of Lanthanide Elements Under Microgravity (Rare Earth Studies 1 & 2)

Many monotectics have already been investigated in space and on Earth, but the influence of the physico-chemical parameters (e.g., atomic volume, heat of evaporation) and kinetic parameters (e.g., gravity, Marangoni convection) on the segregation behavior is not well understood. Because atomic properties of the lanthanide elements change continuously, they are suitable for examining the physico-chemical properties. While mixing of trivalent lanthanides is possible, alloying between trivalent and bivalent lanthanides results in wide mixing gaps. Further, "...bivalent Lanthanides-- under adequate pressure, at high temperatures... can change into the trivalent state during alloying... and can then be mixed completely with other trivalent Lanthanides and related d^1 elements." (1, p. 18, translated)

This TEXUS 13 experiment was designed to determine if the Eu-La system (which exhibits a miscibility gap in the liquid phase) would result in a finely dispersed alloy in a low-gravity environment. (Both width and size of the miscibility gap can be varied over a wide range by a "suited selection" of the alloying elements.) Theorists predicted a critical solution temperature for the Eu-La system to be between 800 and 4700 K. Reportedly, Bach et al. determined the critical solution temperature (experimentally) to be 1235 K (as reported in Reference (7)). <Note: it appears from Reference (6) that the temperature was experimentally determined in the 1-g environment.>

Just prior to the launch of the rocket, six samples (of five different compositions within the miscibility gap) were heated above their critical temperature at a rate of 1 K/s to 1400 ^{o}C. (See the **Sample Materials** section below for a description of each of the five compositions.) During the flight, the samples were quenched at a rate of 9.36 K/s. <Note: The length of time the samples were held at 1400 ^{o}C was not detailed.>

The low-gravity samples were evaluated using raster electron microscopy and energy dispersive X-ray techniques and then compared to Earth-processed reference samples. Reportedly, "For all 5 concentrations we found a clear separation in an enriched phase of Eu and in a homogeneous phase of $Eu_{20}La_{80}$ under 1-g... as well as under microgravity. No finely dispersed solution was observed....

"Under microgravity the melts with concentrations of $Eu_{26}La_{74}$ and $Eu_{30}La_{70}$ remained on the bottom of the crucibles. The melts with concentrations of $Eu_{40}La_{60}$, $Eu_{60}La_{40}$, and $Eu_{65}La_{35}$ were also found on the top of the crucibles dependent on the interfacial tension between crucible and melt which is influenced by the Eu concentration....

"During quenching the crucible wall is cooler than the sample. Therefore the higher melting alloy enriched with La goes to the wall. Small particles of the enriched phase of La are inhomogeneously distributed in the enriched phase of Eu. A free surface or an inhomogeneous temperature profile may cause transport mechanisms (interface convection) which explain this observed inhomogeneous distribution....

"Under 1 g condition the enriched phase of La was found on the bottom of the crucible because of its higher density. Small particles of the enriched phase of La are nearly homogeneously distributed in the enriched phase of Eu." (4, p. 6)

It was concluded that the separation is mainly governed by the differences in interfacical tension and viscosity of the components. Reportedly, therefore, the investigators intend to measure the dependence of these parameters on temperature and concentration in the ternary system Eu-La-Sm.

A more detailed discussion of the experiment can be found in Reference (8).

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Monotectic Compositions, Rare Earth Alloys, Dispersion Alloys, Metallic Matrix, Binary Systems, Model Materials, Melt and Solidification, Phase Separation, Liquid/Liquid Dispersion, Homogeneous Dispersion, Liquid/Liquid Interface, Particle Motion, Segregation, Density Difference, Separation of Components, Free Surface, Interfacial Tension, Marangoni Convection, Viscosity, Thermal Gradient, Solid/Liquid Interface, Quench Process, Wetting of Container, Crucible Effects, Material Interaction With Containment Facility

Number of Samples: Six samples of five different compositions.
Sample Materials: Rare Earth alloys of $Eu_{26}La_{74}$, $Eu_{30}La_{70}$, $Eu_{40}La_{60}$, $Eu_{60}La_{40}$, and $Eu_{65}La_{35}$.
(Eu*La*)
Container Materials: Molybdenum alloy (TZM)
(Mo*)

Experiment/Material Applications:
The investigators regard the lanthanide elements as a metallic model system to better understand the separation behavior in monotectics. It is their goal to determine under what conditions finely dispersed solutions with new or improved properties can be prepared in the space environment.

References/Applicable Publications:
(1) Sprenger, S., Bach, H., and Methfessel, S.: Entmischungsverhalten der Seltenen Erden unter μg- und 1-g Bedingungen. In BMFT/DFVLR TEXUS 13-16 Abschlussberict, 1988, pp. 18-22. (post-flight; in German)

(2) Hüser, D., Römer, R., Bach. H., and Methfessel, S.: Bestimung des Phasendiagramms bon $La_{1-x}Eu_x$ für Experimente unter Mikrogravitation. Verhandl. DPG (VI) 21, 1328 (1986).

(3) Sprenger, S., Bach, H., and Methfessel, S.: Entmischungsverhalten des Systems La-Eu auf der Erde und unter Mikrogravitation. Verhandl. DPG (VII) 22, M-12.3 (1987).

(4) Bach, H., Hüser, D., Methfessel, S., Abd-Elmeguid, M. M., and Sprenger, S.: The Monotectic System Eu-La under 1-g and Low Gravity Condition. (post-flight) <Note: The publication status of this document is unclear at this time.>

(5) Bach, H.: Separation in Monotectics of Lanthanide Elements Under Microgravity. COSPAR Landesbericht, 1988. (post-flight)

(6) Experiment-Modul TEM 01-1. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, p. 17. (processing facility; in German)

(7) Input received from Principal Investigator H. Bach, June 1988.

(8) Sprenger, S., Bach, H., and Methfessel, S.: Segregation Behaviour of Rare Earths Under Microgravity and Normal Gravity Conditions. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 228-233. (post-flight)

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Experiment Origin: Japan
Mission: TEXUS 13
Launch Date/Expt. Date: April 1986
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01: Isothermal four-chamber furnace. (Only one of the chambers was employed for this experiment.)
Builder of Processing Facility: ERNO Raumfahrttecnik GmbH, Bremen, Germany

Experiment:
Fabrication of Superconducting Materials

This TEXUS 13 experiment was designed to study the solidification of an Al-Pb-Bi monotectic alloy. The experiment, which explored the fabrication of superconducting materials, was a preliminary investigation performed prior to, and in support of, the First Materials Processing Test of Japan (FMPT). <Note: The FMPT (equipped with Togano's experiments) later flew on the U.S. shuttle during Spacelab J, in 1992.>

Prior to the rocket launch, an 8.7 mm diameter, 14 mm long Al-6.8 wt.% Pb-6.8 wt.% Bi alloy was configured within a boron-nitride crucible and preheated to 1200 $^{\circ}$C. During the low-gravity phase of the mission, the alloy was solidified.

Post-flight, the TEXUS sample was compared to an Earth-processed sample, heat-treated in the same furnace with the same temperature-time profile. Reportedly, a significant difference in microstructure was observed between Earth-and-flight processed alloys. In the earth-processed alloy, most of the Pb-Bi particles condensed at the bottom of the crucible, indicating that gravity-induced segregation had occurred. In contrast, the flight sample had a much more homogeneous distribution of Pb-Bi alloy particles in the Al matrix. However, there was non-uniformity in size distribution of of Pb-Bi particles in the flight sample; larger particles existed in the upper part of the solidified alloy. Reportedly, this non-uniformity was most likely caused by the migration of Pb-Bi particles along the temperature gradient during the cooling process.

Few additional details concerning this experiment could be located at this time.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Superconductors, Superconductivity, Monotectic Compositions, Ternary Systems, Melt and Solidification, Metallic Matrix, Density Difference, Segregation, Separation of Components, Phase Separation, Homogeneous Dispersion, Liquid/Liquid Dispersion, Particle Dispersion, Liquid/Liquid Interface, Solid/Liquid Interface, Marangoni Movement of Droplets, Sample Microstructure, Particle Size Distribution, Particle Migration

Number of Samples: one
Sample Materials: Al-6.8 wt.% Pb-6.8 wt.% Bi
(Al*Pb*Bi*)
Container Materials: boron nitride crucible contained within a nickel cartridge
(B*N*)

Experiment/Material Applications:
The production of new superconducting materials is investigated in the low-gravity environment where more homogeneous dispersions of an alloy into a metal matrix are expected.

References/Applicable Publications:
(1) Togano, K., Yoshida, Y., Tachikawa, K., and Nii, K.: The Solidification of Superconducting Al-Pb-Bi Alloys Under Microgravity. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 23-26. (in German; post-flight)

(2) Ratke, L.: Immiscible Alloys Under Microgravity Conditions. (discusses TEXUS 13 results) <Note: The publication status of this document is unclear at this time. Reportedly, the document was to be published in Advances In Space Research.>

(3) Togano, K., Yoshida, Y., Tachikawa, K., and Nii, K.: Studies on the Solidification of Superconducting Al-Pb-Bi Alloy Under Microgravity-Results of the TEXUS 13 Mission. In Space 1986, October 16-17, 1986. (post-flight)

(4) Input received from Experiment Investigator, August 1989.

(5) Solidification of an Al-Pb-Bi Alloy Under Microgravity. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 274-277. (post-flight)

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Experiment Origin: Italy
Mission: MASER 1
Launch Date/Expt. Date: March 1987
Launched From: Esrange, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: One high precision isothermal furnace housed within the Multi Mission Mirror Furnace Module (M4)
Builder of Processing Facility: SAAB Space, Sweden, and Swedish Space Corporation, Solna, Sweden

Experiment:
Meniscus Stability in Immiscibles

"A [cited] theoretical model... predicts that liquid bridges formed in microgravity by immiscible metals are stable as long as the ratio L^3/V (L: length and V: volume of the bridge) is kept below a critical value which depends on the wetting angle of the interface on the support plates." (11, p. 278)

This MASER 1 experiment was designed to investigate the stability of liquid bridges formed by two immiscible Zn-Bi fluids. The specific objectives of the research included (1) verifying theoretical stability criteria for (two-liquid) immiscible fluid bridges during the low-gravity phase of the rocket, (2) observing the wetting behavior and measuring the contact angles of a metallic liquid-liquid interface in contact with support plates, and (3) verifying the technical setup of the experiment (which was to be used for other investigations as well).

Five experiment cells were prepared prior to launch and then loaded into one stainless steel sample cartridge. Each cell contained a cylindrical rod of conjugated Zn-Bi (approximately 83 wt.% Bi), vacuum melted and cast axially between two similarly sized cylindrical rods of nearly monotectic Zn-Bi (approximately 5 wt.% Bi). The composite rod was then placed between two support plates such that the longitudinal axis of the rod paralleled the longitudinal axes of the plates. Thus, the plates were in contact with both types of Zn-Bi molten liquids during the experimentation. <Note: A figure detailing the experiment cells can be found in several of the references below (e.g. Reference (11), p. 279)> (See the Sample Materials section below for a detailing

of the five alloy/plate combinations.) The distance between the plates was chosen (based on theoretical calculations) such that three of the cells should have a stable liquid bridge configuration, and two of the cells should have an unstable configuration.

The sample cartridge was placed into a high precision isothermal furnace located within the Multi-Mission Mirror Furnace Module (M4). Three mirror arrays and 30 halogen lamps were configured within the furnace to produce the desired isothermal temperature distribution on the cartridge. Several thermocouples measured the thermal distribution during processing.

Prior to the rocket launch, the cartridge was heated and stabilized to 410 $^{\circ}$C. <Note: It appears that this action melted the middle cylindrical rod in each of the five samples.> During the low-gravity phase of the mission, the samples were heated to 430 $^{\circ}$C. <Note: It appears that this action resulted in the complete melting of the samples.> The temperature stabilized at this value for the next 300 seconds. The sample was then cooled (via nitrogen gas) to 400 $^{\circ}$C 70 seconds before the onset of the rocket-reentry phase. (The principal thermocouple read 380 $^{\circ}$C at the onset of the reentry phase.)

Reportedly, the high-precision isothermal M4 furnace "worked very well" and the technical setup used for this experiment was verified. Metallographic examinations of the five cells were presented and some of the observations are detailed here: "[Generally, in the rocket experimental cells]... some voids are present in specific locations, namely in the central upper part of the cells. This could be due to volume contraction of the Bi-rich phase which had to melt under 1-g conditions, before launch. Similarly, on cooling, the Zn-rich phase contracted during solidification, leaving some room to the Bi-rich phase, still liquid, to penetrate along the ceramic-metal interface giving rise to a pseudo-wetting pattern.

"A homogeneous distribution of Zn needles is seen inside the Bi-rich phase, in all the cells, with no segregation, at variance with ground reference samples." (2, p. 65)

"The trace of a nearly perfect liquid bridge between two silica plates could be observed... [in cell number 2]. This was considered as the first experimental evidence of the possibility to form stable liquid bridges in immiscible metals. However, the bridge profiles were disturbed on cooling and cannot be reliably compared to computed ones." (11, p. 278)

As expected, three of the cells were stable while the remaining two were not, verifying the theoretical estimations of the stability of the bridges. "The stable cells maintained a nearly

axial symmetry whereas... [the unstable cells] appear disturbed and shifted towards the crucible lateral wall." (2, p. 65) It was also noted that the liquid bridges were very sensitive to residual accelerations.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Binary Systems, Metallic Matrix, Monotectic Compositions, Melt and Solidification, Isothermal Processing, Liquid Bridges, Liquid Bridge Stability, Liquid Stability, Meniscus Shape, Meniscus Stability, Liquid/Liquid Interface, Phase Separation, Liquid/Liquid Dispersion, Homogeneous Dispersion, Segregation, Capillary Forces, Wetting, Contact Angle, Solid/Liquid Interface, Needles, Voids, Sample Microstructure, Volume Change, Acceleration Effects, Halogen Lamps

Number of Samples: five
Sample Materials: Cell #1: Zn-Bi alloy/two vitreous silica ceramic plates (the distance between the plates corresponded to an unstable system). Cell #2: Zn-Bi alloy/two vitreous silica plates (the distance between the plates corresponded to a stable system). Cell #3: Zn-Bi alloy/two boron nitride plates (the distance between the plates corresponded to a stable system). Cell #4: Zn-Bi alloy/two graphite plates (the distance between the plates corresponded to a stable system). Cell #5: Zn-Bi alloy/two graphite plates (the distance between the plates corresponded to an unstable system). <Note: See **Experiment** summary above for more information.>
(Zn*Bi*)
Container Materials: Stainless steel (see also the Sample Materials section above for plate materials).

Experiment/Material Applications:
This experiment has applications in both materials processing and fluids research areas. For example, "Many phenomena, such as liquid phase sintering processes, separation of immiscible alloys,... [crystal growth] from molten phases and technical measurements like interfacial energy determination, are strongly related to the shape and stability of the liquid meniscus which sets up between the liquid phases and the solid supports." (5, p. 91)

References/Applicable Publications:

(1) Zaar, J. and Änggard, K.: MASER and Its Effectiveness and Experimental Results. In: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP), 32 pp. (post-flight; short description)

(2) Passerone, A., Rossitto, F., and Sangiorgi, R.: Meniscus Stability in Immiscible Metals-MASER 1 Experiment. Applied Microgravity Technology, 1, 1988, pp. 62-66. (post-flight)

(3) Rossitto, F., Passerone, A., Sangiorgi, R., and Minisini, R.: Liquid Bridges Formed by Immiscible Metals-A Sounding Rocket Experiment. In Proc. 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, pp. 215-220, ESA SP-256. (preflight)

(4) Sangiorgi, R., Muolo, M. L., and Passerone, A.: Wettability of Sintered AlN by Liquid Al and In. Materials Science Monographs, 38A, Elsevier, Austrolem, Part A, 1987. (related research)

(5) Rossitto, F. and Passerone, A.: Stability of Liquid Bridges in Microgravity Conditions. Materials Under Extreme Conditions, MRS Europe, Ed. le Physique, 1985, pp. 91-95. (related research)

(6) Input received from Experiment Investigator, July 1990 and August 1993.

(7) Jönsson, R.: The Microgravity Program in Sweden - Emphasis on the Materials Rocket Maser. In 15th International Symposium on Space Technology and Science, Tokyo, Japan, May 19-23, 1987, pp. 2099-2110. (preflight)

(8) Zaar, J., Björn, L., and Jönsson, R.: Preliminary MASER 1 Results and the Evolution of the MASER Programme. In Proceedings of the 8th ESA Symposium on European Rocket and Balloon Programmes and Related Research, Sunne, Sweden, May 17-23, 1987, ESA SP-270, pp. 359-361. (post-flight; very short description)

(9) Grunditz, H.: Flight Results of the ESA Experiment Modules in MASER 1. In Proceedings of the 8th ESA Symposium on European Rocket and Balloon Programmes and Related Research, Sunne, Sweden May 17-23, 1987, ESA SP-270, pp. 363-367. (post-flight)

(10) Grunditz, H.: Experiment Equipment for Metallurgy and Fluid Science Studies Under Microgravity. 37th Congress of the International Astronautical Federation, Innsbruck, Austria, October 4-11, 1986. (post-flight)

(11) Meniscus Stability in Immiscible Metals. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 278-279. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 15
Launch Date/Expt. Date: May 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1
Builder of Processing Facility: MBB/ERNO, Bremen, Germany

Experiment:
Separation of Monotectic Ternary Alloys

On Earth, alloys which have a monotectic minority phase embedded in a multiphase matrix are often difficult to produce. During processing of such systems, rapid sedimentation of the droplets of the denser minority phase results in the coagulation of the dense phase at the bottom of the sample.

This TEXUS 15 experiment was the first in a series of investigations designed by Prinz et al. to study the separation of monotectic ternary alloys.

Before the rocket flight, three 9.8 mm diameter, 6.7 mm long samples of the Al-Si-Bi monotectic ternary alloy were placed in boron-nitride crucibles and sealed in a Ni-cartridge. Thermocouples were located at the outer surface of the cartridge. Just prior to launch, the cartridge was heated to 470 ^{o}C.

Reportedly, shortly after the successful launch of the TEXUS 15 rocket, data and television transmitters experienced a partial failure. It was discovered that a lateral burn-through of the second stage of the rocket had occurred and the stage, in turn, had collided with the prematurely separated payload. The upper part of the payload including the TEM 01-1 module parachuted to the Earth undamaged.

It appears (from Reference (2)) that during a low-gravity portion of the flight, the sample cartridge temperature was first increased above 950 ^{o}C and then lowered (cooled) by flushing helium at the crucible bottom.

It had been expected (if the rocket failure had not occurred), that the low parity condition during the experiments would have

led to a spatially homogeneous distribution of dispersed Bi-phase in the matrix of Al-primary crystals and Al-Si-eutectic.

Documentation detailing any analysis of the three samples does not appear to be available.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Monotectic Compositions, Ternary Systems, Eutectics, Minority Phase, Multiphase Media, Metallic Matrix, Phase Separation, Melt and Solidification, Sedimentation, Separation of Components, Density Difference, Drops, Coagulation, Homogeneous Dispersion, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Solid/Liquid Interface, Rocket Motion, Acceleration Effects, Payload Survivability, Rocket Failure

Number of Samples: three
Sample Materials: Monotectic ternary alloys: AlSi2.5Bi5, AlSi5Bi5, and AlSi5Bi20 (in wt.%) <Note: Reference (2), which was the only reference which listed the sample materials, presented them in this confusing format.>
(Al*Si*Bi*)
Container Materials: The samples were contained in boron nitride crucibles and sealed in a single Ni-cartridge.
(B*N*, Ni*)

Experiment/Material Applications:
"The Al-based monotectic alloys, where a monotectic minority phase is embedded in a multiphase matrix, are expected to have significantly improved bearing properties." (Reference (2))

References/Applicable Publications:
(1) Experimentelle Nutzlast und Experimente TEXUS 15. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 107-108. (in German; post-flight)

(2) Input received from A. Romero, August 1991 and August 1993.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 16
Launch Date/Expt. Date: November 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1
Builder of Processing Facility: MBB/ERNO, Bremen, Germany

Experiment:
Separation of Monotectic Ternary Alloys

This TEXUS 16 experiment was the second in a series of investigations designed by Prinz et al. to study the separation of monotectic ternary alloys (see Prinz, TEXUS 15). The experimental setup and expected inflight procedure were similar to those described under Prinz, TEXUS 15.

Reportedly, shortly after the successful launch of TEXUS 16, fuel in the second stage of the rocket did not ignite as planned. After the apogee was reached, and the rocket began to fall, the yo-yo despin system was deployed as programmed. Due to the unexpected excess rocket mass, however, there was an incomplete reduction of rocket spin. Subsequently, the payload separated from the second stage, but the parachute was not released. An unbraked impact of the payload resulted in the destruction of all experiment modules including the TEM 01-1 module.

No further discussion of this experiment could be located at this time.

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Alloys, Metallic Matrix, Monotectic Compositions, Ternary Systems, Eutectics, Minority Phase, Phase Separation, Multiphase Media, Melt and Solidification, Sedimentation, Separation of Components, Density Difference, Drops, Coagulation, Dispersion, Homogeneous Dispersion, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Solid/Liquid Interface, Rocket Motion, Acceleration Effects, Payload Survivability, Rocket Failure

Number of Samples: three
Sample Materials: Monotectic ternary alloys: AlSi2.5Bi5, AlSi5Bi5, and AlSi5Bi20. <Note: Reference (2), which was the only reference which listed the sample materials, presented them in this confusing format.>
(Al*Si*Bi*)
Container Materials: The samples were contained in boron nitride crucibles and sealed in a single Ni-cartridge.
(Bi*N*, Ni*)

Experiment/Material Applications:
See Prinz, TEXUS 15.

References/Applicable Publications:
(1) Die Kampagne TEXUS 16. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 109-111. (in German; post-flight)

(2) Input received from A. Romero, August 1991 and August 1993.

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Co-Investigator(s): Unknown
Affiliation(s): (1) Chemistry Department, University of Alabama, Huntsville, Alabama; (2) Department of Math, University of California, Berkeley, California

Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Twelve glass cuvettes (1.5-ml) filled with immiscible fluids. Stirring bars mixed the fluids.
Builder of Processing Facility: Unknown

Experiment:
Demixing of Immiscible Polymers

When appropriate amounts of polymers such as polyethylene glycol (PEG) and dextran are mixed with water on Earth, demixing occurs rapidly and a two-phase, liquid-liquid system results. In this two-phase terrestrial system, most of the (lighter) PEG is located in the top phase and most of the (heavier) dextran is located in the bottom phase.

As detailed in the applications section below, such two-phase demixing is important for several reasons including a biological purification technique which relies on the selective partitioning of the phases. If the two-phase systems will selectively partition in the low-gravity environment, benefits of such low-gravity separation might be (1) reduced biological cell sedimentation and (2) reduced rate of phase emulsion demixing.

A related investigation on the earlier STS 51-D which examined the phase partitioning of two-phase polymer systems (see Brooks, STS 51-D (Chapter 1)) indicated that the systems did demix in the low-gravity environment. Further, the demixing occurred at slower rates and more uniformly than Earth-based separations.

This Consort 1 experiment was the third in a series of investigations designed by Harris and/or Brooks et al. to evaluate the demixing of aqueous polymer two-phase systems (see Brooks, STS 51-D; Brooks, STS-26 (both in Chapter 1)). The major objective of the experiment was to "...determine if the mixing rate and location of the aqueous polymer two-phase systems can be controlled by changing the wall wetting... with polymer coatings on the container and by changing container shape." (2, p. 345)

Prior to the rocket launch, 12 glass cuvettes (each 1.5 ml) were filled with various immiscible liquids. <Note: The following items were not detailed in applicable documents written after the Consort 1 flight: (1) the employed immiscible liquids, (2) the specific coatings applied to the cuvettes and (3) the shape of each of the cuvettes.> The rocket was launched and 15 seconds prior to the initiation of the low gravity phase, liquid pairs in each of the cuvettes were mixed for 30 seconds by a motor-driven stirring bar. The subsequent demixing was photographed every 15 seconds.

Post-flight analysis of the photographs revealed that (1) the film was underexposed and (2) two of the stirring bars did not operate as anticipated. Further, "...the phases which were mixed did not demix to any significant degree during the 7-minute low g period, even though these same systems were largely demixed during the same time on... [the] recent STS-26 flight...." (4, p. 29)

Speculations were presented as to why the demixing did not occur as anticipated. On STS-26, mixing of the fluids was done by hand (shaking of the fluid-filled container which held a mixing ball); while on Consort 1, a more efficient mixing method was used (mechanical stirring bars). Thus, it was suggested that the more efficient mixing on Consort 1 had produced significantly smaller droplet sizes in the fluids systems. Secondly, the corresponding STS-26 experiments were performed at a temperature of 28 ^{o}C, while the Consort experiments were performed at a temperature of 19 ^{o}C. Earlier work had indicated that the demixing occurs more rapidly at higher temperatures. <Note: Specific references to this earlier work were not detailed.>

"The contents of the unstirred cuvettes separated very nicely into two phases: one phase had moved to one side of the cuvette and formed an irregular shape. The shape remained relatively unchanged and motionless during the remainder of the photographs. Densitometry analysis clearly showed the separation.... The seeming lack of motion was an indication of a relatively good low gravity environment on-board the rocket once the low gravity portion of the flight was attained. Previous experiments on the shuttle showed these shapes to be very sensitive to accelerations." (1, p. 8)

Key Words: Systems Exhibiting a Miscibility Gap, Immiscible Fluids, Binary Systems, Polymers, Aqueous Solutions, Multiphase Media, Transparent Liquids, Model Materials, Stirring of Components, Liquid Mixing, Liquid Demixing, Separation of Components, Phase Partitioning, Phase Separation, Liquid/Liquid Interface, Liquid/Liquid Dispersion, Emulsion, Interfacial Tension, Drops, Droplet Size, Drop Migration, Segregation, Sedimentation, Density Difference, Wetting, Wetting of Container, Container Shape, Crucible Effects, Coated Surfaces, Contained Fluids, Biotechnology, Photographic Difficulties, Hardware Malfunction, Acceleration Effects

Number of Samples: twelve
Sample Materials: aqueous two-phase (immiscible) systems
Container Materials: Twelve 1.5 ml glass cuvettes. It appears some (if not all) of the cuvettes may have (1) been coated with Dextran coatings, and/or (2) been of various shapes.

Experiment/Material Applications:
"The interfacial tension in these two-phase [immiscible] systems is very low (approximately a thousand times less than that for a typical organic-water two-phase system) and they can be buffered with various salts. As a consequence, biological materials such as proteins and cells are quite stable in these systems. An important biological purification technique is based on this partitioning of materials between the two phases and the interface.... The systems also serve as transparent fluid models with which to study the fluid physics of demixing processes in polymer blends, polymer-gas foams, and metals. This knowledge is applicable to (1) providing a better understanding of the role of phase segregation and domain size and uniformity in determining the properties of polymer blends and polymer foams... (2) modeling of demixing of immiscible metals... and (3) purification of biological materials by partitioning between the two liquid phases formed in the aqueous polymer two-phase systems." (2, p. 345)

References/Applicable Publications:
(1) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results - A Synopsis. 40th Congress of the International Astronautical Federation, October 7-12, 1989, Màlaga, Spain, IAF-89-439, 11 pp. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (post-flight)

(3) Harris, J. M.: Physical Properties of Immiscible Polymers. In Consortium for Materials Development in Space, Technical Section, pp. 72-77, University of Alabama, Huntsville, Alabama, (1986-1987). (preflight)

(4) Harris, J. M.: Physical Properties of Immiscible Polymers. In 1989 Annual Report of the Consortium for Materials Development in Space, University of Alabama, Huntsville, Alabama, pp. 28-31.

(5) Bamberger, S., Van Alstine, J. M., Harris, J. M., Baird, J. K., Snyder, R. S., Boyce, J., and Brooks, D. E.: Demixing of Aqueous Polymer Two-Phase Systems in Low Gravity. Separation Science and Technology, 23(1-3), pp. 17-34. (preflight; KC-135 results)

(6) Concus, P.: Equilibrium Fluid Interfaces in the Absence of Gravity. Lawrence Berkeley Laboratory, University of California Physics Division, presented at the American Society of Mechanical Engineers, Winter Meeting, Anaheim, California, December 1, 1986. (related research)

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Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: A series of rectangular cavities containing the sample resins; two silicone rubber heaters.
Builder of Processing Facility: Dr. F. C. Wessling, Consortium for Materials Development in Space at the University of Alabama in Huntsville

Experiment:
<u>Elastomer-Modified Epoxy Resins Heater</u>

Pre-Consort experiments (not specifically identified in Reference (2)), which examined the demixing of immiscible liquids in a reduced gravity environment, illustrated that "...the demixing process proceeds unusually slowly to give spherical [droplet] domains undistorted by sedimentation." (2, p. 31) Thus, because it was expected that the "...domain size and morphology will affect the mechanical properties of... [immiscible polymer] blends..." (2, p. 31), experiments were performed on Consort 1 to examine the droplet morphologies and distributions of elastomer modified epoxy resins.

Such resins exist as a single phase until they are heated. When "...heat is applied... catalytic cross-linking begins. As cross-linking proceeds, the elastomer phase separates from the epoxy phase and shortly thereafter the morphology is frozen by solidification." (2, p. 31)

The specific objective of this Consort experiment was "...to examine several of the epoxies with different compositions giving different phase separation points." (2, p. 31) Reportedly, the experimental package consisted of "...a thin mold of aluminum with a series of rectangular cavities containing the resin. This mold... [was] sandwiched by thin aluminum plates which... [were] in turn, sandwiched by two silicone-rubber heaters." (2, p. 31)

During the low-gravity phase of the mission, the package was rapidly heated (within 200 seconds) to 200 ^{o}C. During the next 4 minutes, the 200 ^{o}C temperature was maintained and all 12 resin samples were cured. The temperature was maintained at 200 ^{o}C for 1 minute longer, then the heating was terminated as the rocket exited the low-gravity phase.

Post-flight analysis of the samples was still in progress at the time References (1) and (2) were written. However, it was reported that unexpectedly, all 12 low-gravity samples appeared translucent. (Similarly processed ground-based samples were opaque.) Further, "Preliminary examination indicates that little phase separation occurred during the flight. If phase demixing indeed proceeds more slowly in low-g (as indicated by our other work with immiscible liquids) this would be expected." (2, p. 32) It appears that the resultant deposition of rubber in epoxy was still to be determined by electron microscopy at the time these references were written.

<Note: Previous pre-Consort experiments discussed in the first paragraph of this experiment summary most likely refer to two shuttle experiments performed by Brooks et al. and/or to KC-135 experiments which were related to the Brooks shuttle experiments (see Brooks, STS 51-D, STS-26 (Chapter 1)). The experiment, "Demixing of Immiscible Polymers," is also applicable to this experiment (see Harris, Consort 1 (this chapter).>

<Note: Chapter 5, "Composites With Solid Particles," contains another experiment involving the curing of epoxy resins (see Dalley, STS-004, "Composite Curing").>

Key Words: Systems Exhibiting a Miscibility Gap, Epoxy Resins, Elastomer Modified Epoxy Resins, Two-Phase System, Polymers, Melt and Solidification, Phase Separation, Separation of Components, Liquid Demixing, Sedimentation, Drops, Droplet Dispersion, Particle Distribution, Particle Size Distribution, Curing, Catalysts, Solid/Liquid Interface, Liquid/Liquid Interface

Number of Samples: twelve (four each of three different polymers)
Sample Materials: "The system selected for this experiment consists of three elastomer-modified epoxy resins. The elastomers are commercial products and are of the general class of materials called carboxy-terminated copolymers of butadiene and acrylonitrile (C elastomers). These elastomers can be reacted

with epoxy resins (typically a low molecular weight epoxy resin) to yield an epoxy-capped elastomer."(3, p. 345) "...1 methylimidazole is an effective catalyst for the crosslinking process...." (2, p. 31)

Container Materials: aluminum
(Al*)

Experiment/Material Applications:
It was expected that low-gravity processing would produce samples with differing elastomer droplet morphologies and distributions. If these expectations were realized, an improvement in sample mechanical properties might have resulted. "The addition of rubber [an elastomer] to epoxy lowers the tensile strength and modulus of elasticity...but increases the fracture energy by a factor of 20." (3, p. 345)

References/Applicable Publications:

(1) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results - A Synopsis. 40th Congress of the International Astronautical Federation, October 7-12, 1989, Màlaga, Spain, IAF-89-439, 11 pp. (post-flight)

(2) Elastomer Modified Epoxy Resins. In Consortium for Materials Development in Space, The University of Alabama in Huntsville, Annual Report, Technical Section, October 1, 1988-September 30, 1989, pp. 31-32. (post-flight)

(3) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight)

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CHAPTER 18

TECHNOLOGICAL EXPERIMENTS

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Experiment Origin: USA
Mission: Skylab, SL-2, First Skylab Manned Mission
Launch Date/Expt Date: June 1973 (month experiments were performed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA) area, Skylab Manned Environment
Processing Facility: Exothermic Brazing Package: chamber providing the vacuum of space (part of the Skylab Sphere Forming Experiment, M553)
Builder of Processing Facility: Unknown

Experiment:
Exothermic Brazing (M552)

Brazing is a metal joining procedure in which a braze alloy flows between the surfaces of two metals and solidifies, acting as a cement. The process differs from welding because the base metals (metals to be joined) are not melted. The bond between the base metal and braze alloy is formed by surface alloying: diffusion of the materials across the base-metal/braze-alloy interface forms intermetallic compounds.

The gap between the base metals is filled via capillary action of the braze alloy. The capillary flow of the alloy is affected by (1) the surface tension of the alloy, (2) the width and uniformity of the gap between the base metals, (3) the wetting characteristics of the alloy, and (4) the gravitational forces which may oppose capillary flow.

This Skylab SL-2 experiment was designed to investigate brazing operations under low-gravity conditions. The specific objectives of the study were to (1) simulate the joining of two tubes (via brazing) in space and (2) investigate the low-gravity behavior (mobility, mixing, and capillarity) of a braze alloy.

During the SL-2 mission, the M552 Exothermic Brazing Package was used to conduct the experiment. The experimental hardware was connected to the M512 Materials Processing Facility and used the M512 battery for power. The M552 package contained two nickel and two stainless steel tubes. Each of the four tubes had a 1.9 cm diameter and a 0.12 cm wall thickness. A slit was cut around the perimeter of each of the tubes to simulate two separate tubes

butted against each other. A portion of the perimeter was left uncut to provide support. Surrounding the simulated joint was a sleeve, either nickel or stainless steel, which was brazed to the tube. Between the sleeve and tube were (1) tapered spacer inserts (to provide a specific clearance) and (2) braze alloy rings (71.8 wt.% Ag - 28.0 wt.% Cu - 0.2-0.4 wt.% Li) snapped into grooves near each end of the sleeve. Reportedly, a small portion of the braze rings in the Ni tubes had been irradiated with Ag-110 isotope to allow post-flight mapping of the metal flow patterns.

Each of the four samples was held in its own cylinder which contained (1) fibrous aluminum oxide insulation, (2) an exothermic material (composed of aluminum, boron, titanium dioxide, and vanadium pentoxide), and (3) an ignitor. The entire assembly was held in a chamber that could be evacuated to space.

During the mission, each sample was processed separately over a 2-day period. At the start of an experiment, the chamber was evacuated for 2 hours. Ignition was initiated, resulting in a heating of the exothermic material and melting of the braze alloy. Approximately 90 minutes was required for the complete reaction to occur. The reaction was followed by a 2.75 hour cool-down period. Extensive ground-based experimentation was also performed for comparison. Reportedly, the hardware performed satisfactorily during the mission.

Post-flight examination of the samples included X-ray, autoradiographic, and metallographic techniques (see Reference (1) for details). Results from these investigations led to the following conclusions:

(1) The M552 experiment demonstrated that brazing operations are feasible under low-gravity conditions. The surface tension forces driving capillary flows were dominant during the brazing operation. Examination of the braze alloy distribution demonstrated that dimensional tolerances, particularly gap sizes, were less critical than on Earth. Therefore, "In space fabrication, many joints, which on earth would be produced by welding, should probably be brazed to allow wider fit up tolerances." (1, p. 56)

(2) "The absence of gravity definitely and surprisingly changes the ways in which liquid and solid metals interact. For example, for the same time and temperature conditions of exposure (a) liquid silver-copper alloy dissolves nickel more rapidly in space than on Earth, and (b) solid stainless steel dissolves copper from liquid silver-copper alloy more rapidly in space than on Earth. The detailed mechanisms by which these reactions are hastened have not been positively identified, and this effect of

space environment had not been predicted." (1, p. 56) After preliminary analysis of these results, it was thought that in space, Ni has a higher solubility in a liquid silver-copper alloy than on Earth. However, this higher solubility was later attributed to the more rapid dissolution of Ni under low-gravity conditions. This suggested that (1) saturated liquid metal solutions may be more easily produced and (2) determination of true solubilities would be easier in space.

(3) The low-gravity behavior of (a) the liquid-vapor boundary and (b) the surface tension driven flow of liquid metal is in good agreement with the theoretical predictions.

(4) The addition of the radioisotope tracer to the two Ni samples provided a unique picture of (a) the thermal history of the braze melting and (b) the braze alloy flow pattern. These results indicated an unexpected, complete circumferential mixing of the isotope that was attributed to (a) liquid-state diffusion and/or (b) turbulence in the capillary flow. On Earth, the isotope tended to settle due to gravity-induced sedimentation.

(5) It appeared that gravity, or the lack of gravity, had no effect on the mechanism of alloy solidification. Microstructural details (e.g., dendritic structure, eutectic structure) were the same in the space and Earth samples.

(6) The low-gravity samples contained fewer and smaller shrinkage defects than ground-based samples. This result indicated that gravity-induced forces significantly affect capillary flow on the braze alloy.

(7) The Skylab samples contained less oxide buildup than the Earth-processed materials indicating the adequacy of the space vacuum for brazing operations.

Additional details of the experimental results can be located in Reference (1).

Key Words: Technological Experiments, Brazing, Gap Filling, Melt and Solidification, Metals, Intermetallics, Diffusion, Solubility, Saturated Solution, Radioactive Tracer Diffusion, Isotopes, Binary Systems, Ternary Systems, Alloys, Space Vacuum, Thermocapillary Flow, Capillary Flow, Turbulent Flow, Liquid Mixing, Meniscus Shape, Surface Energy, Surface Tension, Wetting, Solid/Liquid Interface, Liquid/Vapor Interface, Dendritic Structure, Sample Microstructure, Defects, Tracer Material

Number of Samples: four
Sample Materials: Samples 1 and 2 (joint material): pure nickel; samples 2 and 3 (joint material): stainless steel. Samples 1 and 2 (braze alloy): 72 wt.% Ag-28 wt.% Cu-0.2wt.% Li with 110-Ag radioactive tracer isotope; samples 3 and 4 (braze alloy): 72 wt.% Ag-28 wt.% Cu-0.2wt.% Li
(Ni*Ag*, Ag*Cu*Li*)
Container Materials: not applicable

Experiment/Material Applications:
Brazing is a process which may be used for repairing or building large structures in space.

The specific reasons why Ni and stainless steel tubes were chosen for this brazing experiment were not presented in available literature.

References/Applicable Publications:

(1) Williams, J. R.: Skylab Experiment M552 Exothermic Brazing. In Proceedings of the Third Space Processing Symposium on Skylab Results, April 30-May 1, 1974, Marshall Space Flight Center, Huntsville, Alabama, Vol. 1, June 1974, pp. 33-84. (post-flight)

(2) Tobin, M. and Kossowsky, R.: Research Study on Materials Processing in Space Expt. M512, Final Report on M551, M552 and M553. NASA CR-120479, 1973. (post-flight)

(3) Bourgeois, S.: Convection Effects on Skylab Experiments M551, M552, and M553. Phase C Report, NASA CR-120482, 1973. (preflight)

(4) Pattee, H. E. and Monroe, R. W.: Characterization of Exothermic Brazing Components, Skylab Experiment M552. NASA CR-12518, 1973. (post-flight)

(5) Heine, R. W., Adams, C. M., and Siewert, T. A.: Flight/Ground Sample Comparison Relating to Flight Experiment M552, Exothermic Brazing. NASA CR-12059, 1973. (post-flight)

(6) Braski, D. N., Adair, H. L., and Kobisk, E. H.: Radioactive Tracer Studies in the NASA Skylab Exothermic Brazing Experiment M552. NASA CR-129035, 1974. (post-flight)

(7) Muraki, R. and Masubichi, K.: Discipline Report on Thermal Analysis of M551, M552 and M553 Experiments. NASA CR-120513, 1974. (post-flight)

(8) Larson, D. J., Jr.: Metallurgical Analysis of Skylab M552 and 558 Samples, Final Report. Grumman Research Dept., Report RE-565, 1978. (post-flight)

(9) Bourgeois, S. V.: Convection Effects on Skylab Experiments M551, M552, and M553-Phase C Report. LMSC-HREC TR D 306955, Lockheed Missiles and Space Company, Huntsville, Alabama, December 1, 1973. (post-flight)

(10) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements during Skylab)

(11) "Experiment M552-Exothermic Brazing," In MSFC Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1984, pp. 5-31 - 5-34. (post-flight)

(12) "M512 Materials Processing Facility," In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-1 - 5-18. (processing facility)

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Experiment Origin: Federal Republic of Germany/Austria
Mission: TEXUS 2
Launch Date/Expt. Date: November 1978
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: Metallurgical Isothermal Heating Facility (IHF) Furnace
Builder of Processing Facility: Unknown, probably Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Capillary Brazing

This TEXUS 2 experiment was the first in a series of investigations designed by Bathke and/or Frieler et al. to evaluate the effects of low-gravity on the vacuum brazing cycle. Of particular interest was the resultant reduced-gravity capillary-driven flow of the braze alloy into gaps between the base material. It was expected that this flow would not be influenced by the weight of the melts.

The TEXUS specimen consisted of an assembly of four nickel cylinders nestled one inside the other. The outer cylinders formed a 200 μm concentric cylindrical gap, while the inner cylinders formed a 0-2000 μm variable clearance sickle-shaped gap. Prior to the flight, the whole assembly was housed in a stainless steel cartridge and sealed after evacuation to an internal pressure of less than 10^{-2} Pa.

During the mission, the assembly was brazed in a resistance heated furnace with a near-eutectic Ag-Cu alloy. The alloy contained trace amounts of Li (which promoted spreading) and radioactive Ag (which acted as a tracer). As indicated by system thermocouples, both Earth and low-gravity processed specimens did not reach the desired peak temperature in all locations. "Therefore, the 200 μm gaps of both specimen[sic] were filled only partially in the shorter portion, the bulk of the braze remaining in the depot region." (1, p. 120)

Post-flight, specimens were evaluated by X-ray radiography, autoradiography, photography and metallography techniques. Reportedly, "In principle the same type of microstructure [of the

Ni/AgCu system] could be observed in both 1-g and low-gravity specimens.... However, the distribution of various phases showed marked differences between these two specimens." (1, p. 118) (The TEXUS sample exhibited an increased precipitation of the primary phase which was attributed to the low-gravity reduced mass transport.)

It was noted that under low-gravity conditions, "...gaps with a width of up to 2000 μm can be filled due to the action of capillary forces." (1, p. 120) "However, the shrinkage porosity increased in the largest area of the brazing seam." (7, pp. 352) Because both 1-g and low-gravity samples illustrated that "...circumferential flow of the braze did occur inside the depot prior to gap filling... [and] radiation could be detected in every part of the filled gap..." (1, p. 120), it was concluded that "...transport mechanisms responsible for the mixing are independent of gravity." (1, p. 120)

Key Words: Technological Experiments, Brazing, Gap Filling, Diffusion, Radioactive Tracer Diffusion, Melt and Solidification, Mass Transfer, Metals, Eutectics, Alloys, Ternary Systems, Thermocapillary Flow, Capillary Forces, Capillary Flow, Surface Tension, Free Surface, Liquid Spreading, Wetting, Liquid Mixing, Hydrostatic Pressure, Tracer Material, Precipitation, Porosity, Vacuum, Processing Difficulties

Number of Samples: one
Sample Materials: An assembly of thin-walled pure nickel tubing forming annular gaps (nickel purity 99.6 wt.%). Filler material: near-eutectic alloy of 71.7 wt.% Ag, 28 wt.% Cu, and 0.3 wt.% Li. (The filler alloy contained radioactive Ag to act as a tracer.) (Ni*), (Ag*Cu*Li*), (Ag*)
Container Materials: The whole assembly was housed in a stainless steel cartridge.

Experiment/Material Applications:
Vacuum brazing is frequently employed to join metals. "However, investigations of brazing reactions and mechanisms remain largely empirical because of the multitude of base metal/filler metal multicomponent systems and the multiplicity of the interactions of numerous essential parameters." (1, p. 117) Details of the brazing process and the effects of gravity on gap filling were investigated here.

References/Applicable Publications:
(1) Freiler, K., Philippovich, N., Stickler, R., and Bathke, W.: Capillary Brazing Under Microgravity (Texus-II) and 1G Conditions. Advances in Space Research, Vol. 1, No. 5, 1981, pp. 117-120. (post-flight)

(2) Schlecker, H. and Szimkowiak, P.: Preparation, Implementation and Evaluation of Material Science Experiments Under the SLN Program TEXUS 2. In TSLP Final Report January 1980, RPT# BMFT-FB-W-81-033. (in German)

(3) Bathke, W., Philippovich, N., Stickler, R., and Frieler, K.: Brazing of Capillary Gaps. In Shuttle/Spacelab Utilization Final Report Project TEXUS II, DFVLR, Köln, 1978, pp. 62-90. (post-flight)

(4) Philippovich, N., Frieler, K., Stickler, R., and Bathke, W.: Brazing Under Microgravity- TEXUS II Experiment. In ESA 3rd European Symposium on Materials Sciences in Space, Grenoble, April 24-27, 1979, ESA SP-142, pp. 95-100.

(5) Frieler, K., Stickler, R., and Siegfried, E.: Brazing Under Microgravity in a Resistance Heated Furnace. Materials Processing in the Reduced Gravity Environment of Space, Materials Research Society Symposia Proceedings, Vol. 9, pp. 639-649.

(6) Siegfried, E., and Frieler, K.: Vakuumlöten unter Mikrogravitation. In Status Seminar 1981 des Bundesministerium für Forschung und Technologie, Spacelab-Nutzung, Werkstofforschung und Verfahrenstechnik im Weltraum, 1981, DGLR-Bericht 81-01, pp. 213-220.

(7) Brazing of Capillary Gaps. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 352-353. (post-flight)

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Experiment Origin: Austria
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Material Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility (IHF) Furnace
Builder of Processing Facility: Entwicklingsring Nord (ERNO), Bremen, Germany

Experiment:
Vacuum Brazing 1ES305/1ES304

This Spacelab 1 experiment was the second in a series of investigations designed by Frieler and/or Bathke et al. to evaluate the effects of low-gravity on the vacuum brazing cycle (see Bathke, TEXUS 2 (this chapter)). The specific objectives of the experiment were to evaluate (1) gap filling mechanisms, (2) movement of a filler metal within a braze depot by means of a tracer metal, and (3) evolution of the microstructure of brazed gaps.

The flight specimen consisted of an assembly of concentrically arranged (hollow) nickel cylinders. The assembly formed a variety of gap geometries and were referenced as (1) the multigap sample (MSP), (2) the annular gap sample (RSP), and (3) the sickle-shaped sample (SSP).

During the mission, the assembly was brazed in the Spacelab Isothermal Heating Facility (IHF) with a near-eutectic AgCu alloy. The alloy contained (1) a small quantity of Li (to promote spreading of the filler material) and (2) 60 wt.% Au, 20 wt.% Ag and 20 wt.% Cu (to act as a tracer material). First the assembly was heated to a temperature slightly less than the melting point of the filler material. After a thermal equilibrium was established in the sample, the assembly was heated past the fill material melting temperature and then cooled.

Reference samples (identically processed on the ground) were compared to the flight sample. More vivid mixing was seen in the flight sample, and it was thought that surface tension gradients were responsible for the enhanced mixing. "As observed already in the earlier TEXUS II experiment... considerable mixing of the

molten braze occurs along the specimen's circumference." (1, p. 96) While all gaps in the 1-g sample were filled, only the outer 200 μm gap of the low-g sample was filled completely. "This unexpected result is attributed to a difference in heat flow between the 1-g and low-gravity specimen. In the 1-g case the hydrostatic pressure provides a constant contact of the molten braze with the inner cylinders of the MSP and thus a means of heat transport through the liquid. In the low-gravity case an... [annular] void was formed within the molten braze. This prevented a contact of the front ends of the gaps with the liquid and so impeded the heat transport by means other than radiation." (1, p. 96)

An evaluation of the microstructure indicated that increased porosity was observed in the low-gravity samples. This porosity was due "...to the fact that bubbles created by instabilities or outgassing processes are not removed by buoyancy forces." (1, p. 98) Sample evaluation also indicated that appreciable segregation of the melt and CuNi dendrites took place in the 1-g sample, while no such segregation was seen in the low-gravity sample.

In summary, results were: (1) that the microstructure is independent of the gravitational level (as opposed to the findings of other investigators), (2) that certain gap geometries will necessarily lead to filling defects under low-gravity conditions, and (3) that there is convection occurring within the braze depot during filling.

Key Words: Technological Experiments, Brazing, Gap Filling, Diffusion, Melt and Solidification, Heat and Mass Transfer, Alloys, Metals, Ternary Systems, Thermocapillary Flow, Thermocapillary Convection, Absence of Buoyancy Forces (Detrimental), Liquid Mixing, Capillary Forces, Capillary Flow, Contact Angle, Hydrostatic Pressure, Surface Tension, Free Surface, Liquid Spreading, Wetting, Segregation, Porosity, Sample Microstructure, Dendrites, Bubbles, Outgassing, Tracer Material, Vacuum, Thermal Equilibrium

Number of Samples: one flight specimen with a variety of gap geometries
Sample Materials: An assembly of nickel tubing was used to create the gaps (base metal 99.6 wt.% Ni with traces of C,Cu,Fe,Mn,S,Si,Cr,Ti,Co). Filler Metal: Near Eutectic AgCu with traces of Li. (71.81 Ag, 28.02 Cu, 0.148 Li). Tracer metal: 60 wt.% Au, 20 wt.% Ag, 20 wt.% Cu.
(Ni*C*Cu*Fe*Mn*S*Si*Cr*Ti*Co*), (Ag*Cu*Li*), (Au*Ag*Cu*)

Container Materials: Standard IHF cartridge. (The specific cartridge material was not indicated.)

Experiment/Material Applications:
Reportedly, the chosen materials are by themselves, of no significance. The aim of the experiment was to investigate the process of brazing per se. Space processing was desirable in order to suppress the effects of hydrostatic pressure and thermal convection.

References/Applicable Publications:

(1) Frieler, K., Stickler, R., and Siegfried, E.: Vacuum Brazing Under Microgravity. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 95-99. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. Teledyne Brown Engineering publication (acceleration measurements on Spacelab 1)

(3) Whittmann, K: The Isothermal Heating Facility. In 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 49-54. (IHF processing facility information)

(4) Frieler, K. and Stickler, R.: Hartlöten unter Weltraumbedingungen, Endbericht über Experiment SL-305 im Rahmen von SL-1. 87-IB-SL-B1, Institut für Physikalische Chemie der Universität Wien, Wien, 1987. (final report on ES305)

(5) Input received from Principal Investigator K. Frieler, July 1988.

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Experiment Origin: Sweden
Mission: MASER 2
Launch Date/Expt. Date: February 1988
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Multi-Mission Mirror Furnace Module (M4). (One of two available isothermal mirror furnaces in the M4 was used for this experiment. The furnace employed three linear-elliptical mirror arrays.)
Builder of Processing Facility: Saab Space, Linköping, Sweden, and The Swedish Space Corporation, Solna, Sweden

Experiment:
Adhesion of Metals on Ceramic Substrates (Direct Determination of Wetting by Molten Metals)

The efficiency of the brazing of ceramic materials with metals depends greatly on the wetting capability of the materials. The adhesion characteristics of the braze can often be improved by adding chemically active metals to the braze.

This MASER 2 experiment was designed to investigate the wetting of the brazing alloy CuAgTi on alumina, zirconia, titanium nitride, and Incoloy (a Ni alloy). The specific objectives were to (1) "...investigate the component distribution in the braze, [(2)] ...determine the attractive forces between the braze and the substrate and [(3)]... assess the influence of the surface roughness on the wetting behaviour of the braze." (5, p. 356)

The experiment was one of two investigations performed within the MASER Multi-Mission Mirror Furnace Module (M4) during the mission (see also Fredriksson, MASER 2, "A Study of the Coalescence process of Immiscible Alloys in Large Samples" (Chapter 17)).

The M4 contained two identical isothermal mirror furnaces, one of which was used for this investigation. The furnace was "...equipped with three linear-elliptical mirror arrays and each array...[was] furnished with a set of ten halogen lamps. These lamps...[were] individually controlled by a microcomputer to give the correct temperature profile on the sample..." (2, p. 13)

<Note: It is unclear (1) how many samples were employed during the investigation and (2) how these samples were individually configured within the single isothermal furnace.>

It was reported (Reference (5)) that "Drops of Cu-Ag alloys of both hypo- and hypereutectic compositions (65 to 85 wt% Ag) and with addition of 2 wt% of Ti were processed in titanium nitride, alumina, zirconia and incoloy cells (6 mm diameter, 7 mm high), whose walls presented different surface roughness. All cells were stacked up in a single cartridge which was processed in an isothermal furnace at 900 ^{o}C for 300 s under microgravity conditions." (5, p. 356)

<Note: In contrast to Reference (5), Reference (4) reported that two different concentrations of Ti in the CuAgTi brazing alloy were examined (2 and 5 wt%).>

It was reported that (1) the M4 operated essentially as expected, (2) controlled heating and cooling of the samples were achieved, and (3) all samples were processed as planned.

<Note: The few results that were presented were somewhat difficult to understand without further details of the experiment setup and inflight melting and solidification procedure. The results are presented below as they appeared in References (4) and (5).>

"Due to the spinning of the rocket during the launch, the drops were located in the corners of the cells. Once melted they were submitted to a dominating capillary force." (5, p. 356) "We found that the molten metal drops were aligned in the same way in all ceramic cells which shows that even the microgravity force is sufficient enough to overcome surface attractive forces, at least during the initiation of wetting. A very pronounced tendency of capillary penetration could also be observed which suggests a beneficial exploitation of this effect in engineering designs...." (4, Appendix 4, p. 1) ("On earth, the alloys were enriched in Cu opposite to gravity and consequently, presented an asymmetrical Ti-build up at the metal/ceramic interface...." (5, p. 356))

When discussing only the Ti 2 wt.% samples it was reported that "The component distribution in the alloys processed in microgravity was uniform... independent of the asymmetry of the substrate and of its roughness. The interfaces were uniformly covered with Ti, thus yielding an even coupling. A better adhesion on alumina was observed in microgravity.... The wetting angle increased with the roughness... and this effect was more pronounced when the adhesion was weaker, as for instance with alumina." (5, p. 356)

<Note: Reference (4) reported that "While with 5 wt% Ti all the wetting experiments were successful, in some cases the 2 wt% content resulted in bad wetting. This appeared both on TiN and ZrO_2

substrates." (4, Appendix 1, p. 1)>

"A significant conclusion can be drawn already at this stage of evaluation with regard to the mechanism of wetting with active braze. It is active only if it can react with the substrate oxide or nitride forming an intermediate coupling layer. Being deposited on metallic substrate (Incoloy) without chance to undergo the above reactions it could not bring about wetting at all. (4, Appendix 4, p. 1)

It was concluded that (1) in general, better wetting was achieved under low gravity conditions than at 1-g conditions, and (2) "...the interfacial segregation of Ti which is of prime importance for the mechanical strength of brazed joints can be more accurately adjusted and the influence of the alloy composition can be better studied in a microgravity environment." (5, p. 356)

Very little additional information concerning this experiment could be located at this time.

Key Words: Technological Experiments, Brazing, Adhesion of Metals, Melt and Solidification, Binary Systems, Ternary Systems, Metals, Alloys, Hypoeutectics, Hypereutectics, Drops, Substrates, Vapor Deposition, Ceramics, Wetting, Wetting of Container, Wetting Kinetics, Contact Angle, Free Surface, Surface Tension, Capillary Forces, Thermocapillary Flow, Liquid Spreading, Mass Transfer, Meniscus Shape, Segregation, Isothermal Processing, Solid/Liquid Interface, Liquid/Gas Interface, Interface Physics, Mechanical Strength, Acceleration Effects, Rocket Motion, Rotation of Payload, Launch-Induced Fluid Motion, Surface Roughness, Oxidation, Halogen Lamps

Number of Samples: unclear
Sample Materials: Braze metal: (1) CuAgTi (2 wt% Ti), (2) CuAgTi (5 wt% Ti); substrate materials: (1) alumina, (2) zirconia, (3) titanium nitride, and (4) Incoloy
(Cu*Ag*Ti*, Al*O*, Zr*O*, Ti*N*)
Container Materials: unclear

Experiment/Material Applications:
"Adhesive bonding has an ever increasing application for the production of structural joints. The advantages over the common mechanical joining (riveting, welding) such as simpler design,

increased fatigue and corrosion resistance, and reduced costs are sufficient to explain the growth of interest in the use of adhesively bonded structures." (3, p. 860)

Although not specifically stated in the available publications, it appears this experiment was performed in the reduced gravity environment to achieve a more uniform distribution of Ti at the solidification interface (with less segregation than is possible in 1-g) and thus improve the mechanical strength of brazed alloy.

References/Applicable Publications:

(1) Zaar, J.: MASER and Its Effectiveness and Experimental Results. In: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center, 32 pp. (preflight; very short description)

(2) Zaar, J. and Dreier, L.: MASER 2 Final Report, RMLO1-7, August 30, 1988, Swedish Space Corporation, including Appendix 4 and 5. (post-flight)

(3) Kozma, L. and Olefjord, I.: Basic Processes of Surface Preparation and Bond Formation of Adhesively Joined Aluminum. Materials Science and Technology, October 1987, Vol. 3, Number 10. (no space results; details adhesive bonding of aluminum techniques)

(4) Kozma, L.: Preliminary Report on the MASER-2 Experiment: "Adhesion of Metals on Ceramic Substrates." In MASER 2 Final Report, RMLO/1-7, August 30, 1988, Swedish Space Corporation, Appendix 4 (and 5). (post-flight)

(5) Adhesion of Metals on Ceramic Substrates. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 356-357. (post-flight)

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Experiment Origin: Sweden
Mission: TEXUS 5
Launch Date/Expt. Date: April 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Ellipsoidal mirror furnace
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Soldering of Sn-Ag between Cu Tubes

Early low-gravity brazing experiments, which were performed to investigate the filling of nickel tubing by AgCuLi (see Williams, Skylab SL-2, and Bathke, TEXUS 2 (this chapter)) indicated that while (1) capillary filling of wide gaps was enhanced in the low-gravity environment, (2) "...increased dissolution of the base material... or gravity independent flow... occurred." (1, p. 337)

Reference (1) reported that the evaluation of the SL-2 and TEXUS 2 results was complicated because (1) the braze-alloy/base-metal system had not been "fully investigated" at the time the experiments were performed, and (2) only limited control of the temperature cycle was possible. <Note: The specific meaning of "fully investigated" as used above was not clear to the editors.>

"The objectives of the [TEXUS 5] experiments were to investigate whether the improved gap filling also occurs during soldering and to carefully... [analyze] the influence of temperature cycle on dissolution and solidification in narrow gaps." (1, p. 337) Reportedly, a "well-known" solder/base-metal system (SnAg-Cu) was employed for the experiments.

Prior to the mission, three samples were prepared. Each sample was designed to permit the study of capillary spreading of Sn-Ag solder between Cu-tubes. The first sample had a cylindrical inner section, resulting in a gap of even width (0.5 mm). The second sample had a conical inner section in which the gap increased in width from 0.2 mm to 0.7 mm. The third sample had a conical inner section in which the gap increased from 0.2 to 1.0 mm.

During the mission, the samples were to be heated in an ellipsoidal mirror furnace to a temperature beyond the solder melting point, and allowed to cool. <Note: It is not clear if all three samples were heated as expected. While (1) Reference (1) reported that the first and second samples were heated with the intended temperature profiles but the third was not heated as expected, (2) Reference (4) did not indicate that any of the samples were incorrectly processed.> The flight samples were compared to similarly processed ground-based samples.

The following results were reported:
(a) "...in the cylindrical sample...nearly all the solder contained in the reservoir was sucked into the gap.... This was not the case in the ground processed samples.
[(b)] the conical sample presenting a gap from 0.20 mm to 0.70 mm was evenly filled up to a width of 0.56 mm....The conical sample where the gap increased from 0.20 mm to 1.0 mm was filled up to 0.58 mm....

"It was concluded that a maximum gap width of close to 0.6 mm can be filled with the system Cu/Sn-Ag. In the corresponding reference samples processed on Earth, the gaps were never uniformly filled up to this width.

"The space and ground samples exhibited different microstructures, particularly with respect to the volume fraction of the Cu-Sn phase. But this could be interpreted as the effect of slightly different temperature/time profiles." (4, p. 354)

It was concluded that the TEXUS low-gravity environment promoted "...the capillary filling of wider gaps during soldering in the system Sn-Ag-Cu.... [Theoretical and experimental analyses indicated]...that small differences in the temperature cycle had a strong influence on the interaction between the solder and the base material during dissolution and solidification. Any influence on the dissolution due to convection could not be revealed." (1, p. 342)

(A more lengthy discussion of the experiment results can be found in Reference (1).)

Key Words: Technological Experiments, Soldering, Melt and Solidification, Surface Tension, Gap Filling, Thermocapillary Flow, Capillary Forces, Capillary Flow, Liquid Spreading, Wetting, Flux, Dissolution, Convection, Ternary Systems, Alloys, Metals, Solid/Liquid Interface, Sample Microstructure, Processing Difficulties

Number of Samples: three
Sample Materials: Copper tubes; filler material: tin-silver solder. "The fluxing agent Zn Cl in water solution, was applied to the inner surfaces of the samples before the inner parts of the samples were pushed into the outer tubes." (1, p. 337)
(Cu*, Sn*Ag*, Zn*Cl*)
Container Materials: unknown

Experiment/Material Applications:
Experiments employing the "well-known" Sn-Ag-Cu system were designed to increase the knowledge of capillary flow and soldering under reduced gravity.

References/Applicable Publications:
(1) Carlberg, T. and Liljendahl, M.: Soldering Under Microgravity. In Proceedings of the 4th European Symposium on Material Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, pp. 337-342.

(2) Carlberg, T., Fredriksson, H., Sunnerkranz, P-A., Grahn, S., and Stenmark, L.: The Swedish TEXUS Experiment - A Technical Description and Some Preliminary Results. Esrange Symposium (1978), ESA SP-135, pp. 325-338. (experimental furnace facility)

(3) Input received from Experiment Investigator, May 1988.

(4) Soldering in Microgravity. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 354-355. (post-flight)

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Experiment Origin: USA
Mission: STS Launch #4, STS-004 (STS OFT-4 Columbia)
Launch Date/Expt. Date: June 1982
Launched From: NASA Kennedy Space Center, Florida
Payload Type: College Student Experiment
NASA Get Away Special (GAS) Canister G-001
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-001: Utah State University, Logan, Utah/R. Gilbert Moore
Processing Facility: Four separate heating and temperature monitoring units
Builder of Processing Facility: Designed and constructed by Principal Investigator G. C. Alford at Utah State University, Logan, Utah

Experiment:
Soldering (Experiment Number 11-M)

This experiment was one of ten investigations housed within the G-001 Get Away Special Canister during STS-004. Four other experiments (of the ten) were applicable to this data base (see Dalley, STS-004 (Chapter 5); Elwell, STS-004 (Chapter 12); Laher, STS-004 (Chapter 17); and Thomas, T. L., STS-004 (Chapter 14)).

The soldering experiment was the first in a series of investigations designed by Alford et al. to study the separation of flux from solder. Investigators suspected that in a low-gravity environment, the "...lack of buoyancy could allow pockets of flux to become trapped in the solder and significantly lower the reliability of a joint by reducing its mechanical strength and electrical conductivity." (1, p. 12)

Ninety-six samples of resin core and coreless solder were to be melted during the mission. "The samples initially project from a 3.5 X 2.0 inch printed circuit board. A spring behind the PC board feeds the solder as it melts into the heated foil." (1, p. 12) The resulting flight samples were to be compared to ground-based solder samples.

Reportedly, experiment objectives could not be completed as planned. Data recorder failure coupled with experiment fuse blowout disrupted the expected experiment procedures.

No further details of this investigation could be located at this time.

Key Words: Technological Experiments, Soldering, Flux, Melt and Solidification, Absence of Buoyancy Forces (Detrimental), Separation of Components, Material Strength, Electrical Conductivity, Surface Tension, Wetting, Solid/Liquid Interface, Hardware Malfunction

Number of Samples: 96
Sample Materials: resin core and coreless solder
Container Material: The solder was melted on flat heated copper foil sheets.
(Cu*)

Experiment/Material Applications:
A knowledge of low-gravity solder and flux separation characteristics will be required during in-orbit replacement of dislodged or damaged electronic components. This experiment was formulated to study the resultant mechanical strength and electrical conductivity of space-produced solder joints.

References/Applicable Publications:
(1) Yoel, D., Walker, S., Elwell, J., and Moore, G: The First Getaway Special-How it was Done. Spaceworld, May 1983, pp. 9-16. (post-flight)

(2) STS-4 Fourth Space Shuttle Mission, NASA Press Kit, June 1982, p. 62. (preflight)

(3) Yoel, D. W.: Payload Integration of a Get Away Special Canister. American Institute of Aeronautics and Astronautics, Annual Meeting and Technical Display on Frontiers of Achievement, Long Beach, California, May 12-14, 1981, 5 pp. (preflight)

(4) The STS-4 Getaway Special. NASA Report PB82-10223, May 20, 1982. (preflight)

(5) Cargo Systems Manual: Gas STS-4. May 20, 1982, JSC-17645, pp. 4-1 - 4-4. (preflight; very short description)

(6) Overbye, D.: The Getaway Kids Shuttle Into History. Discover, September 1982. (post-flight)

(7) Yoel, D. W.: Analysis of the First Getaway Special Space Shuttle Payload. Thesis for M.S. in Physics, Utah State University, Logan, Utah, 1984. (post-flight)

(8) Moore, R. G.: Educational Implications of Getaway Special Payload Number One. IAF-81-293, XXXIInd International Astronautical Federation Congress, Rome, September 6, 1981. (preflight)

(9) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(10) "Get Away Special," NASA News, NASA MSFC, June 7, 1982.

(11) Transcripts of press conference at NASA MSFC with G-001 Student Experimenters and Sponsors, NASA, May 20, 1982.

(12) Input received from Principal Investigator G. C. Alford, July 1993.

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Co-Investigator(s): Megill, L. R. (Payload Manager) (2), Utah Section of American Institute of Aeronautics and Astronautics (Purchaser and Donor) (3)
Affiliation(s): (1) Prior to STS-011: Utah State University, Logan, Utah, Currently: Uniden Corporation of America, Ft. Worth, Texas; (2) During STS-011: Utah State University Faculty, Logan, Utah, Currently: ARME Enterprises, Hyrum, Utah; (3) Utah

Experiment Origin: USA
Mission: STS Launch #10, STS-011 (STS 41-B Challenger)
Launch Date/Expt. Date: February 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: College Student Experiment
NASA Get Away Special (GAS) Canister G-008
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
(This experiment was a reflight of a soldering experiment previously flown on STS-004 in G-001.)
Primary Developer/Sponsor of this experiment within G-008: Utah State University, Logan, Utah
Processing Facility: Solder, heating elements, thin metal plates
Builder of Processing Facility: Principal Investigator, G. C. Alford

Experiment:
Soldering Experiment

This experiment was one of four investigations housed within the G-008 Get Away Special Canister during STS-011. Two other experiments (of the four) were applicable to this data base (see Gerpheide, STS-011 (Chapter 16); Walden, STS-011 (Chapter 18)). The soldering experiment was the second in a series of investigations designed by Alford et al. to study the separation of flux from solder (see Alford, STS-004). The specific objective of the experiment was to examine the solder/flux separation characteristics of space-produced solder joints.

The Principal Investigator reported the following: "Approximately 3 g of SN 60 solder was melted... and resolidified on two flat, heated copper plates. The solder was in the form of 62 individual samples. The samples were a mixture of solid, cored, and multi-cored solder wire, and solder paste. The solder wires were fed linearly into the copper plates as the wires melted. The solder paste was divided into sixteen individual circular samples. Each paste sample was applied directly to the copper plates. The copper plate that the coreless solder was melted to was pre-fluxed.

"Post-flight inspection of the samples for trapped pockets of flux... was done with an industrial X-ray microscope. As expected, no trapped flux was found in the micro-gravity melted coreless solder. A small percentage of the cored sample melts contained trapped flux. Most of the multi-core samples contained some trapped flux. The solder paste samples exhibited only partial melting. Near the heat source, the solder alloy formed spherical globes within the flux paste. The diameter of the globes became progressively smaller as distance from the heat source increased. It is not known whether these spherical globes would have coalesced into a single mass if more heat had been applied. Significantly, solder from the paste did wet portions of the copper foil on which it was melted." (Reference (7))

No other details concerning this experiment could be found.

Key Words: Technological Experiments, Soldering, Flux, Melt and Solidification, Absence of Buoyancy Forces (Detrimental), Separation of Components, Material Strength, Electrical Conductivity, Surface Tension, Wetting, Solid/Liquid Interface

Number of Samples: 62
Sample Materials: resin core, multi-core and coreless solder and solder paste
Container Material: The solder was melted on flat heated copper sheets.
(Cu*)

Experiment/Material Applications:
See Alford, STS-004.

References/Applicable Publications:

(1) Cargo Systems Manual: GAS Annex for STS-11, JSC-17645 Annex STS-11, December 2, 1983. (preflight, very short description)

(2) Getaway Special (GAS) Payloads (STS-11). In Goddard Space Flight Center's Engineering Newsletter, Vol. 2, No. 3, April 1984, published by the Engineering Directorate, pp. 8-9. (very short description)

(3) STS-11 Getaway Special Payload Descriptions, NASA News, NASA GSFC, 1984. (preflight)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(5) STS 41-B, Tenth Space Shuttle Mission, Press Kit, February 1984, p. 28. (brief mention of experiment; preflight)

(6) STS-11 GAS Payloads. NASA Goddard Space Flight Center Engineering Newsletter, April 1984.

(7) Input received from Principal Investigator G. C. Alford, August 1993.

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Experiment Origin: USA
Mission: STS Launch #6, STS-006 (STS 31-B, Challenger)
Launch Date/Expt. Date: April 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: College Student Experiment
NASA Get Away Special (GAS) canister G-049
  Volume of Canister 5.0 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-049: The United States Air Force Academy, Colorado Springs, Colorado
Processing Facility: A heating tool which melted solder at the connecting points of two beams.
Builder of Processing Facility: Students at the United States Air Force Academy, Colorado Springs, Colorado

Experiment:
Metal Beam Joiner

This experiment was one of six investigations housed within the G-049 Get Away Special canister on STS-006. Four other experiments (of the six) were applicable to this data base (see Amidon, STS-006 (Chapter 14); Neel, STS-006 (Chapter 4); Peter, STS-006 (Chapter 18); Streb, STS-006 (Chapter 14)). The objective of this experiment was to demonstrate the soldering of beams in a low-gravity environment.

Few details of the experiment were available (and the descriptions of the experimental setup, as presented in References (1) and (2), were somewhat unclear). It appears that during the experiment a "63/37" mixture of tin/lead solder (located at the connection point of a male-ended 1/4-inch brass beam and a female-ended 1/4-inch brass beam) was melted using a 48 watt (16 volt) wire wrapped heating tool. Upon hardening, the solder joined the two 1/4-inch beams together.

Post-flight analysis of the connection indicated that the solder had bonded the two beams together.

Bond strength tests were performed on beams which had been soldered on the ground and these results related to the space produced bond (the single flight bond did not actually undergo tension tests): "In 1983 an Air Force Laboratory performed non-destructive testing by comparing X-rays of the space beam and 12 ground soldered beams to check for significant voids. The space beam fell within the range of ground-soldered results. With only a single sample, we made no statement as to the statistical significance of the result." (6)

<Note: It appears based on this result, the space bond was expected to be able to withstand well over 174 kg of tension.>

More detailed information concerning the experiment could not be located at this time.

Key Words: Technological Experiments, Soldering, Welding, Melt and Solidification, Material Strength, Surface Tension, Wetting, Solid/Liquid Interface

Number of Samples: two beams
Sample Materials: 1/4-inch brass beams joined with a "67/37" mixture of lead-tin solder
(Cu*Zn*, Pb*Sn*)
Container Materials: unknown

Experiment/Material Applications:
Although this experiment used solder to form a bond between two beams, the investigators indicated that the research concept could be extended for future welding endeavors. Construction of space structures (or the repair of existing structures) may require welding of materials in the low-gravity, space environment.

References/Applicable Publications:
(1) Cargo Systems Manual: GAS Annex for STS-6, JSC-17645 Annex STS-6, December 3, 1982. (very short summary; preflight)

(2) Swan, P. and Worsowicz, C.: The Eaglets Have Flown. Space Education, Vol. 1, No. 7, May 1984, pp. 317-319. (post-flight)

(3) STS-6 Getaway Specials. NASA News, NASA GSFC, November 24, 1982. (preflight)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(5) NASA STS-6 Sixth Space Shuttle Mission Press Kit, April 1983, pp. 41-43. (preflight)

(6) Input received from Principal Investigator H. G. Gross, August 1993.

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Co-Investigator(s): Fortune, W. S. (President, EDSYN, Inc.) (2)
Affiliation(s): (1) Van Nuys, California; (2) EDSYN, Inc., Van Nuys, California

Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) canister G-088
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-088: EDSYN, Inc., Van Nuys, California
Processing Facility: System consisting of (1) a printed circuit board/copper clad assembly, (2) heater, and (3) solid-core solder
Builder of Processing Facility: Unknown, probably: EDSYN, Inc., Van Nuys, California

Experiment:
Solder Flux Selection Test (Dynamic Experiment #1)

This experiment was one of nine solder-related investigations contained within the G-088 Get Away Special canister during the STS-007 mission (see EDSYN, STS-007 to review the other eight experiments). The objective of this particular experiment (designated as Dynamic Experiment #1) was to determine which type of flux was best suited for use during low-gravity soldering.

Assembly of the experiment cell took place prior to flight. During this assembly, a circular section of "copper clad," divided into four equal quadrants, was attached to a larger-diameter circular section of printed circuit board. Each quadrant on the clad was coated with a different flux material. The clad/board assembly was mounted against a heater. Solid-core solder, which was wrapped around the heater, was spring loaded at one end against the clad.

The experiment cell was placed within a sealed, hollow aluminum tube. A filter at one end of the tube was configured to collect debris created during the experiment; a view glass at the other end was configured to collect vapor samples resulting from the experimental process. Alkaline dioxide primary cell batteries provided power for the heater.

The experiment cell was configured within Get Away Special canister G-088. Prior to the STS-007 flight, the canister was evacuated to a 1 Torr vacuum level (a vacuum similar to that of

the space environment).

During the experiment, as the solder was melted, the spring-loading arrangement forced the solder onto the copper. Flow properties and wetting characteristics of the flux-coated clad were observed.

A ground-based reference experiment was performed for comparison. Photographs of the clad/heater assembly were taken before and after the ground-based experiments were performed.

Reportedly, during the space experiment, the solder unexpectedly "...bunched up at the end of the heater... where it was forced by the spring...." (1, p. 7) Very little solder flowed onto the clad. The flux on all four quadrants melted "...and the fumes were deposited on the inside of the tube, on the view glass and in the filter. The quantity of deposits was much thicker than expected." (1, p. 7)

During the ground-based experiment, the solder melted, flowed onto the clad, and spread over the copper. Gravity and surface wetting forces were responsible for the resultant fluid flow.

No further information concerning the space results of Dynamic Experiment #1 appear to be available at this time.

Key Words: Technological Experiments, Soldering, Flux, Melt and Solidification, Wetting, Surface Tension, Liquid Spreading, Coated Surfaces, Solid/Liquid Interface, Vaporization, Vapor Deposition, Vacuum

Number of Samples: one demonstration unit
Sample Materials: (1) solid-core solder, (2) four different flux materials, (3) copper clad, and (4) printed circuit board
(Cu*)
Container Materials: A sealed, hollow aluminum tube
(Al*)

Experiment/Material Applications:
The soldering-related experiments were performed to (1) examine the physics of solder alloying and (2) identify complications arising from debris and fume production in a low-gravity (or vacuum) environment.

The research, which is related to spacecraft repairs or other repair tasks, was performed in a low-gravity, vacuum environment. Thus, it applies to repairs performed in the (outer) space environment rather than to repairs performed within the pressurized vehicle environment.

References/Applicable Publications:

(1) EDSYN NASA Payload No. 88, STS-7. Preliminary report, available from EDSYN Soldering Products, Inc., Van Nuys, California, 18 pp. (post-flight)

(2) "STS-7 Cargo Systems Manual: GAS," JSC-17645 Annex 7 Basic Version PCN-1, NASA JSC, April 1, 1983. (very short description; preflight)

(3) "STS-7 Getaway Specials," NASA News, NASA GSFC, May 1983.

(4) NASA STS-7 Seventh Space Shuttle Mission Press Kit, June 1983, p. 56. (preflight)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special Canister mission history)

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Affiliation(s): (1) Van Nuys, California; (2) EDSYN, Inc., Van Nuys, California

Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) canister G-088
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-088: EDSYN, Inc., Van Nuys, California
Processing Facility: Four copper wires attached to a heating element. Each copper wire was wrapped with solid-core solder and coated with flux.
Builder of Processing Facility: Unknown, probably: EDSYN, Inc., Van Nuys, California

Experiment:
Solder Wetting and Surface Tension I (Dynamic Experiment #2)

This experiment was one of nine soldering-related investigations contained within the G-088 Get Away Special canister during the STS-007 mission (see EDSYN, STS-007 to review the other eight experiments). The objective of this particular experiment (designated as Dynamic Experiment #2) was to determine the wetting and surface tension characteristics of solder on copper wire.

Assembly of the experiment cell took place prior to the flight. During this assembly, four pieces of copper wire were each bent into a circle and attached to a cylindrical heating element. The four circles were configured at different locations around the heating element such that together they formed three log-tapered wire gaps. Solid-core solder, coated with a "mildly activated flux," was wrapped around each of the four wire circles.

The experiment cell was contained within a sealed, hollow aluminum tube. A filter at one end of the tube was configured to collect debris created during the experiment; a view glass at the other end was configured to collect vapor samples resulting from the experiment process. Alkaline dioxide primary cell batteries provided power for the cylindrical heating element.

The experiment cell was configured within Get Away Special canister G-088. Prior to flight, the canister was evacuated to a 1 Torr vacuum level (a vacuum similar to that of the space

environment).

During the space experiment, the solder was melted and solder flow characteristics over the copper wire observed. A ground-based reference experiment was performed for comparison. Photographs of the heater/circle assembly were taken before and after the space- and ground-based experiments were performed.

Reportedly, during the space experiment, the solder bridged the wire gaps and formed a single, very large, lumpy sphere between two of the wire circles.

During the ground-based experiment, the solder flowed around the wires. No bridging of the wire gaps occurred. The gravity component acting on the system forced excess solder to accumulate on the wire circle oriented closest to the ground. The excess solder solidified on this bottom wire (at the point on the circle closest to the ground) in the shape of a sphere.

After all nine experiments within the canister were evaluated, it was concluded that (1) if proper precautions are taken, the control of solder debris is not a significant problem, (2) "Molten solder flowed along the solder path with almost all of it adhering to the item being soldered, and (3) Conversely[,] solder flux is difficult to control since when heated it vaporizes and generates fumes." (1, p. 16)

Key Words: Technological Experiments, Soldering, Flux, Melt and Solidification, Wetting, Surface Tension, Surface Tension Minimum, Liquid Spreading, Coated Surfaces, Solid/Liquid Interface, Vaporization, Vacuum

Number of Samples: one demonstration unit
Sample Materials: solid-core solder, four copper wires, flux
(Cu*)
Container Materials: a sealed, hollow aluminum tube
(Al*)

Experiment/Material Applications:
The soldering-related experiments were performed to (1) examine the physics of solder alloying and (2) identify complications arising from debris and fume production in a low-gravity (or

vacuum) environment.

The research, which is related to spacecraft repairs or other repair tasks, was performed in a low-gravity, vacuum environment. Thus, it applies to repairs performed in the (outer) space environment rather than to repairs within the pressurized vehicle environment.

References/Applicable Publications:

(1) EDSYN NASA Payload No. 88, STS-7. Preliminary report, available from EDSYN Soldering Products, Inc., Van Nuys, California, 18 pp. (post-flight)

(2) "STS-7 Cargo Systems Manual: GAS," JSC-17645 Annex 7 Basic Version PCN-1, NASA JSC, April 1, 1983. (very short description; preflight)

(3) "STS-7 Getaway Specials," NASA News, NASA GSFC, May 1983.

(4) NASA STS-7 Seventh Space Shuttle Mission Press Kit, June 1983, p. 56. (preflight)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

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Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) canister G-088
Volume of Canister: 2.5 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-088: EDSYN, Inc., Van Nuys, California
Processing Facility: A section of (1) round wire, (2) flat wire, and (3) copper braid attached to a solder tip. Each section was wrapped with solid-core solder and coated with flux.
Builder of Processing Facility: Unknown, probably: EDSYN, Inc., Van Nuys, California

Experiment:
Wetting and Surface Tension II: Solder Flow and Bridging Test (Dynamic Experiment #3)

This experiment was one of nine solder-related investigations contained within the G-088 Get Away Special canister during the STS-007 mission (see EDSYN, STS-007 to review the other eight experiments). The objective of this particular experiment (designated as Dynamic Experiment #3) was to determine "...the wetting and surface tension characteristics that relate to the ability of solder [1] to bridge gaps and [2] to flow." (1, p. 9)

Assembly of the experiment cell took place prior to flight. During this assembly, a section of copper braid, a section of round wire, and a section of flat wire were each wrapped with solid-core solder and then coated with flux. All three sections were then attached to one soldering tip at different locations. The round wire was attached perpendicular to the longitudinal axis of the tip; the braid was attached parallel to the longitudinal axis of the tip (out the top of the tip); the flat wire was attached perpendicular to the longitudinal axis of the tip, 180^{o} from the round wire. <Note: It is not clear if the round and flat wires were made of copper.>

The experiment cell was placed within a sealed, hollow aluminum tube. A filter at one end of the tube was configured to collect debris created during the experiment; a view glass at the other end was configured to collect vapor samples resulting from the process. Alkaline dioxide primary cell batteries provided power

for the solder tip.

The experiment cell was configured within Get Away Special canister G-088. Prior to flight, the canister was evacuated to a 1 Torr vacuum level (a vacuum similar to that of the space environment).

During the experiment, the solder was melted. Wetting and flow characteristics were examined.

A ground-based reference experiment was performed for comparison. Photographs of the solder-tip/solder-section assembly were taken before and after the space- and ground-based experiments were performed.

Reportedly, during the space experiment, "Solder flowed out onto three surfaces in a smooth even manner with excess solder covering the entire length of the braid. All surfaces showed excellent wetting." (1, p. 9)

During the ground-based experiment, the solder essentially "...flowed the same on all three surfaces, but flowed further on the copper braid." (1, p. 9) <Note: The orientation of the solder-tip/solder-section assembly with respect to gravity was not detailed in Reference (1).>

After all nine experiments within the canister were evaluated, it was concluded that (1) if proper precautions are taken, the control of solder debris is not a significant problem, (2) "Molten solder flowed along the solder path with almost all of it adhering to the item being soldered, and (3) Conversely[,] solder flux is difficult to control since when heated it vaporizes and generates fumes." (1, p. 16)

Key Words: Technological Experiments, Soldering, Flux, Melt and Solidification, Gap Filling, Wetting, Surface Tension, Liquid Spreading, Solid/Liquid Interface, Coated Surfaces, Vaporization, Vacuum

Number of Samples: one demonstration unit
Sample Materials: solid-core solder, flux, wire, copper braid (Cu*)
Container Materials: A sealed, hollow aluminum tube (Al*)

Experiment/Material Applications:
The type of copper braid employed in the experiment is commonly used for electrical grounding or solder removal.

The soldering-related experiments were performed to (1) examine the physics of solder alloying and (2) identify complications arising from debris and fume production in a low-gravity (or vacuum) environment.

The research, which is related to spacecraft repairs or other repair tasks, was performed in a low gravity, vacuum environment. Thus, it applies to repairs performed in the (outer) space environment rather than to repairs performed within the pressurized vehicle environment.

References/Applicable Publications:
(1) EDSYN NASA Payload No. 88, STS-7. Preliminary report, available from EDSYN Soldering Products, Inc., Van Nuys, California, 18 pp. (post-flight)

(2) "STS-7 Cargo Systems Manual: GAS," JSC-17645 Annex 7 Basic Version PCN-1, NASA JSC, April 1 1983. (very short description; preflight)

(3) "STS-7 Getaway Specials," NASA News, NASA GSFC, May 1983.

(4) NASA STS-7 Seventh Space Shuttle Mission Press Kit, June 1983, p. 56. (preflight)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

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Affiliation(s): (1) Van Nuys, California; (2) EDSYN, Inc., Van Nuys, California

Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) canister G-088
Volume of Canister: 2.5 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-088: EDSYN, Inc., Van Nuys, California
Processing Facility: Eyelet and turret solder post attached to the end of soldering tip
Builder of Processing Facility: Unknown, probably EDSYN, Inc., Van Nuys, California

Experiment:
Metallurgical Properties: Eyelet and Post Soldering Test (Dynamic Experiment #4)

This experiment was one of nine solder-related investigations contained within the G-088 Get Away Special canister during the STS-007 mission (see EDSYN, STS-007 to review the other eight experiments). The objective of this particular experiment (designated as Dynamic Experiment #4) was to observe solder flow into an eyelet and onto a turret solder post.

Assembly of the experiment cell took place prior to flight. During the assembly, an eyelet and post were attached to the end of a soldering tip.

The experiment cell was contained within a sealed, hollow aluminum tube. A filter at one end of the tube was configured to collect debris created during the experiment; a view glass at the other end was configured to collect vapor samples resulting from the experimental process. Alkaline dioxide primary cell batteries provided power to the soldering tip.

The experiment cell was configured within Get Away Special canister G-088. Prior to the STS-007 flight, the canister was evacuated to a 1 Torr vacuum level (a vacuum similar to that of the space environment).

During the experiment, the solder was melted. <Note: The initial solder placement was not detailed in Reference (1).>

A ground-based reference experiment was performed for comparison purposes. Photographs of the soldering tip assembly were taken before and after the space- and ground-based experiments were performed.

Reportedly, during the space experiment, "The solder melted and flowed evenly around the turret post. The eyelet filled with solder and formed a raised area of solder on both sides of the eyelet. (convex) (unexpected)." (1, p. 10)

During the ground-based experiment, "Solder flowed evenly onto the two sections. The turret post soldered normally and the hole simulating an eyelet filled with solder. The solder showed a slight depression (concave) on the top side of the eyelet due to normal surface tension and gravity." (1, p. 10)

After all nine experiments within the canister were evaluated, it was concluded that (1) if proper precautions are taken, the control of solder debris is not a significant problem, (2) "Molten solder flowed along the solder path with almost all of it adhering to the item being soldered, and (3) Conversely[,] solder flux is difficult to control since when heated it vaporizes and generates fumes." (1, p. 16)

Key Words: Technological Experiments, Soldering, Melt and Solidification, Gap Filling, Wetting, Surface Tension, Liquid Spreading, Solid/Liquid Interface, Vaporization, Vacuum

Number of Samples: one demonstration unit
Sample Materials: solder, eyelet, turret solder post (specific materials were unspecified)
Container Materials: a sealed, hollow aluminum tube
(Al*)

Experiment/Material Applications:
The soldering-related experiments were performed to (1) examine the physics of solder alloying and (2) identify implications arising from debris and fume production in a low-gravity (or vacuum) environment.

The research, which is related to spacecraft repairs or other repair tasks, was performed in a low gravity, vacuum environment. Thus, it applies to repairs performed in the (outer) space environment rather than to repairs performed within the pressurized vehicle environment.

Eyelets and turret solder posts are wire attachment points often employed in circuit assemblies.

References/Applicable Publications:

(1) EDSYN NASA Payload No. 88, STS-7. Preliminary report, available from EDSYN Soldering Products, Inc., Van Nuys, California, 18 pp. (post-flight)

(2) "STS-7 Cargo Systems Manual: GAS," JSC-17645 Annex 7 Basic Version PCN-1, NASA JSC, April 1, 1983. (very short description; preflight)

(3) "STS-7 Getaway Specials," NASA News, NASA GSFC, May 1983.

(4) NASA STS-7 Seventh Space Shuttle Mission Press Kit, June 1983, p. 56. (preflight)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

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Co-Investigator(s): Fortune, W. S. (President, EDSYN, Inc.) (2)
Affiliation(s): (1) Van Nuys, California; (2) EDSYN, Inc., Van Nuys, California

Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) canister G-088
Volume of Canister: 2.5 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-088: EDSYN, Inc., Van Nuys, California
Processing Facility: Integrated circuit/printed circuit board assembly
Builder of Processing Facility: Unknown, probably EDSYN, Inc., Van Nuys, California

Experiment:
Solder Removal: Integrated Circuit Removal Test
(Dynamic Experiment #5)

This experiment was one of nine solder-related investigations contained within the G-088 Get Away Special canister during the STS-007 mission (see EDSYN, STS-007 to review the other eight experiments). The objectives of this particular experiment (designated as Dynamic Experiment #5) were to (1) remove an integrated circuit from a printed circuit board, and (2) determine the quantity of debris resulting from the removal. The circuit was to be extracted by a non-conventional method.

Assembly of the experiment cell took place prior to flight. During the assembly, it appears that a spring was compressed under the integrated circuit during soldering of the component to the board. Apparently, when the attachment solder was melted, the circuit was to be pushed away from the board by the spring.

The experiment cell was contained within a sealed, hollow aluminum tube. A filter at one end of the tube was configured to collect debris created during the experiment; a view glass at the other end was configured to collect vapor samples resulting from the experimental process. Alkaline dioxide primary cell batteries provided power for the solder melting.

The experiment cell was configured within Get Away Special canister G-088. Prior to STS-007 flight, the canister was evacuated to a 1 Torr vacuum level (a vacuum similar to that of the space environment).

A ground-based reference experiment was performed for comparison. Photographs of the circuit/board assembly were taken before and after the space- and ground-based experiments were performed.

Reportedly, during the space experiment, "The solder did not melt properly due to lower than expected temperatures inside the payload." (1, p. 11)

During the ground-based experiment, "The solder melted, the spring pulled the integrated circuit out of the circuit board and the solder debris fell into the bottom of the tube." (1, p. 11)

No additional information concerning Dynamic Experiment #5 could be located at this time.

Key Words: Technological Experiments, Solder Removal, Melt and Solidification, Solid/Liquid Interface, Vaporization, Vacuum, Thermal Environment More Extreme Than Predicted, Sample Not Processed as Planned

Number of Samples: one demonstration unit
Sample Materials: solder, integrated circuit, printed circuit board, spring
Container Materials: a sealed, hollow aluminum tube
(Al*)

Experiment/Material Applications:
The soldering-related experiments were performed to (1) examine the physics of solder alloying and (2) identify complications arising from debris and fume production in a low-gravity (or vacuum) environment.

The research, which is related to spacecraft repairs or other repair tasks, was performed in a low gravity, vacuum environment. Thus, it applies to repairs performed in the (outer) space environment rather than to repairs performed within the pressurized vehicle environment.

References/Applicable Publications:
(1) EDSYN NASA Payload No. 88, STS-7. Preliminary report, available from EDSYN Soldering Products, Inc., Van Nuys, California, 18 pp. (post-flight)

(2) "STS-7 Cargo Systems Manual: GAS," JSC-17645 Annex 7 Basic Version PCN-1, NASA JSC, April 1, 1983. (very short description; preflight)

(3) "STS-7 Getaway Specials," NASA News, NASA GSFC, May 1983.

(4) NASA STS-7 Seventh Space Shuttle Mission Press Kit, June 1983, p. 56. (preflight)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

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Affiliation(s): (1) Van Nuys, California; (2) EDSYN, Inc., Van Nuys, California

Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) canister G-088
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-088: EDSYN, Inc., Van Nuys, California
Processing Facility: A pressure chamber connected to a metal tube was attached to a hollow soldering tool.
Builder of Processing Facility: Unknown, probably EDSYN, Inc., Van Nuys, California

Experiment:
<u>Desoldering II: Pressure Desoldering Demo (Dynamic Experiment #6)</u>

On Earth, excess molten solder is usually removed by suction. If solder removal is to be achieved in the space vacuum environment, however, an alternate method must be employed.

This experiment was one of nine solder-related investigations contained within the G-088 Get Away Special canister during the STS-007 mission (see EDSYN, STS-007 to review the other eight experiments). The objective of this particular experiment (designated as Dynamic Experiment #6) was to determine if pressure could be employed to remove excess solder.

Assembly of the experiment cell took place prior to flight. During this assembly, a small pressure chamber connected to a metal tube was attached to a hollow soldering tool. The soldering tool had a hole through its tip. It appears that a heater surrounded the soldering tool. When the tip was heated to the solder's melting point during the experiment, the pressurized air was to be released and the solder blown from the hole.

The experiment cell was contained within a sealed, hollow aluminum tube. A filter at one end of the tube was configured to collect debris created during the experiment; a view glass at the other end was configured to collect vapor samples resulting from the process. Alkaline dioxide primary cell batteries provided power for the heater.

The experiment cell was configured within Get Away Special canister G-088. Prior to the STS-007 flight, the canister was evacuated to a 1 Torr vacuum level (a vacuum similar to that of the space environment).

A ground-based reference experiment was performed for comparison. Photographs of the soldering tool assembly were taken before and after the space- and ground-based experiments were performed.

Reportedly, during the space experiment, the solder melted and all of the solder was blown to the end of the experiment container.

During the ground-based experiment, the solder melted and some of the solder fell to the bottom of the experiment container; the rest of the solder was blown to the end of the container.

After all nine experiments within the canister were evaluated, it was concluded that (1) if proper precautions are taken, the control of solder debris is not a significant problem, (2) "Molten solder flowed along the solder path with almost all of it adhering to the item being soldered, and (3) Conversely[,] solder flux is difficult to control since when heated it vaporizes and generates fumes." (1, p. 16)

Key Words: Technological Experiments, Solder Removal, Melt and Solidification, Gas Pressure, Solid/Liquid Interface, Vaporization, Vacuum

Number of Samples: one demonstration unit
Sample Materials: solder
Container Materials: a sealed, hollow aluminum tube

Experiment/Material Applications:
The soldering-related experiments were performed to (1) examine the physics of solder alloying and (2) identify complications arising from debris and fume production in a low-gravity (or vacuum) environment.

The research, which is related to spacecraft repairs or other repair tasks, was performed in a low-gravity, vacuum environment. Thus, it applies to repairs performed in the (outer) space en-

vironment rather than to repairs performed within the pressurized vehicle environment.

EDSYN has a product which employs hot air for solder removal rather than suction. This experiment demonstrated the solder removal via a method other than the suction procedure.

References/Applicable Publications:

(1) EDSYN NASA Payload No. 88, STS-7. Preliminary report, available from EDSYN Soldering Products, Inc., Van Nuys, California, 18 pp. (post-flight)

(2) "STS-7 Cargo Systems Manual: GAS," JSC-17645 Annex 7 Basic Version PCN-1, NASA JSC, April 1, 1983. (very short description; preflight)

(3) "STS-7 Getaway Specials," NASA News, NASA GSFC, May 1983.

(4) NASA STS-7 Seventh Space Shuttle Mission Press Kit, June 1983, p. 56. (preflight)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

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Affiliation(s): (1) Van Nuys, California; (2) EDSYN, Inc., Van Nuys, California

Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) canister G-088
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-088: EDSYN, Inc., Van Nuys, California
Processing Facility: EDSYN, Inc., Loner$_{TM}$ temperature controlled soldering tool
Builder of Processing Facility: EDSYN, Inc., Van Nuys, California

Experiment:
Electronically Controlled Solder Tip Demo with Flux (Dynamic Experiment #7)

This experiment was one of nine solder-related investigations contained within the G-088 Get Away Special canister during the STS-007 mission (see EDSYN, STS-007 to review the other eight experiments). The objective of this particular experiment (designated as Dynamic Experiment #7) was to verify the operation of an EDSYN, Inc., Loner$_{TM}$ temperature-controlled soldering tool in a low-gravity, vacuum environment.

The electronic temperature control circuitry of this standard model tool was not modified for the experiment. Flux was used to improve solder flow. <Note: The initial placement of the solder was not detailed in Reference (1).>

The soldering tool with solder was contained within a sealed, hollow aluminum tube. A filter at one end of the tube was configured to collect debris created during the experiment; a view glass at the other end was configured to collect vapor samples resulting from the experimental process. Alkaline dioxide primary cell batteries provided power for the soldering tool.

The experiment cell was configured within Get Away Special canister G-088. Prior to flight, the canister was evacuated to a 1 Torr vacuum level (a vacuum similar to that of the space environment). <Note: Additional details of the in-flight experimental setup and procedure were not presented in the available documents.>

A ground-based reference experiment was performed for comparison. Photographs of the soldering tool assembly were taken before and after the space- and ground-based experiments were performed.

Reportedly, during the space experiment, the control circuit "...operated properly and solder melted normally." (1, p. 13)

During the ground-based experiment, the control circuitry operated properly. The "...solder flowed normally and coated uniformly." (1, p. 13)

After all nine experiments within the canister were evaluated, it was concluded that (1) if proper precautions are taken, the control of solder debris is not a significant problem, (2) "Molten solder flowed along the solder path with almost all of it adhering to the item being soldered, and (3) Conversely[,] solder flux is difficult to control since when heated it vaporizes and generates fumes." (1, p. 16)

No additional information concerning Dynamic Experiemnt #7 could be located at this time.

Key Words: Technological Experiments, Soldering, Flux, Soldering-Related Tools, Melt and Solidification, Wetting, Surface Tension, Liquid Spreading, Solid/Liquid Interface, Vaporization, Vacuum

Number of Samples: one demonstration unit
Sample Materials: solder
Container Materials: a sealed, hollow aluminum tube
(Al*)

Experiment/Material Applications:
The soldering-related experiments were performed to (1) examine the physics of solder alloying and (2) identify complications arising from debris and fume production in a low-gravity (or vacuum) environment.

The research, which is related to spacecraft repairs or other repair tasks, was performed in a low-gravity, vacuum environment. Thus, it applies to repairs performed in the (outer) space environment rather than to repairs performed within the pressurized vehicle environment.

This experiment tested the operation of a standard (off-the-shelf) temperature-controlled soldering tool in the low-gravity, vacuum environment.

References/Applicable Publications:

(1) EDSYN NASA Payload No. 88, STS-7. Preliminary report, available from EDSYN Soldering Products, Inc., Van Nuys, California, 18 pp. (post-flight)

(2) "STS-7 Cargo Systems Manual: GAS," JSC-17645 Annex 7 Basic Version PCN-1, NASA JSC, April 1, 1983. (very short description; preflight)

(3) "STS-7 Getaway Specials," NASA News, NASA GSFC, May 1983.

(4) NASA STS-7 Seventh Space Shuttle Mission Press Kit, June 1983, p. 56. (preflight)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

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Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) canister G-088
Volume of Canister: 2.5 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-088: EDSYN, Inc., Van Nuys, California
Processing Facility: EDSYN, Inc., Loner$_{TM}$ temperature controlled soldering tool
Builder of Processing Facility: EDSYN, Inc., Van Nuys, California

Experiment:
Electronically Controlled Solder Tip Demo Without Flux (Dynamic Experiment #8)

This experiment was one of nine solder-related investigations contained within the G-088 Get Away Special canister during the STS-007 mission (see EDSYN, STS-007 to review the other eight experiments). The objective of this particular experiment (designated as Dynamic Experiment #8) was to verify the operation of an EDSYN, Inc., Loner$_{TM}$ temperature-controlled soldering tool in a low-gravity, vacuum environment. (The electronic temperature-control circuitry of this standard model soldering tool was not modified for the experiment.)

Dynamic Experiment #8 was very similar to Dynamic Experiment #7 (see EDSYN, STS-007, Dynamic Experiment #7) except that during Experiment #8, no flux was employed to aid the solder flow. <Note: Initial placement of the solder was not detailed in Reference (1).>

The STS-007 soldering tool with solder was contained within a sealed, hollow aluminum tube. A filter at one end of the tube was configured to collect debris created during the experiment; a view glass at the other end was configured to collect vapor samples resulting from the process. Alkaline dioxide primary cell batteries provided power for the soldering tool.

The experiment cell was configured within Get Away Special canister G-088. Prior to flight, the canister was evacuated to a 1 Torr vacuum level (a vacuum similar to that of the space environment).

A ground-based reference experiment was performed for comparison. Photographs of the soldering tool assembly were taken before and after the space- and ground-based experiments were performed.

Reportedly, during the space experiment, "The solder melted slightly but did not adhere." (1, p. 13)

During the ground-based experiment, "The solder melted, adhered poorly to the tip and hung over the side of the tip." (1, p. 13)

Additional information concerning Dynamic Experiment #8 could not be located.

Key Words: Technological Experiments, Soldering, Soldering-Related Tools, Melt and Solidification, Wetting, Surface Tension, Liquid Spreading, Solid/Liquid Interface, Vaporization, Vacuum

Number of Samples: one demonstration unit
Sample Materials: solder
Container Materials: a sealed, hollow aluminum tube
(Al*)

Experiment/Material Applications:
The soldering-related experiments were performed to (1) examine the physics of solder alloying and (2) identify complications arising from debris and fume production in a low-gravity (or vacuum) environment.

The research, which is related to spacecraft repairs or other repair tasks, was performed in a low-gravity, vacuum environment. Thus, it applies to repairs performed in the (outer) space environment rather than to repairs performed within the pressurized vehicle environment.

This experiment tested the operation of a standard (off-the-shelf) temperature-controlled soldering tool in the low-gravity, vacuum environment.

References/Applicable Publications:

(1) EDSYN NASA Payload No. 88, STS-7. Preliminary report, available from EDSYN Soldering Products, Inc., Van Nuys, California, 18 pp. (post-flight)

(2) "STS-7 Cargo Systems Manual: GAS," JSC-17645 Annex 7 Basic Version PCN-1, NASA JSC, April 1, 1983. (very short description; preflight)

(3) "STS-7 Getaway Specials," NASA News, NASA GSFC, May 1983.

(4) NASA STS-7 Seventh Space Shuttle Mission Press Kit, June 1983, p. 56. (preflight)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

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Affiliation(s): (1) Van Nuys, California; (2) EDSYN, Inc., Van Nuys, California

Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) canister G-088
Volume of Canister: 2.5 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-088: EDSYN, Inc., Van Nuys, California
Processing Facility: None, passive experiment
Builder of Processing Facility: Not Applicable

Experiment:
Product Exposure Test (Experiment #9)

This experiment was one of nine solder-related investigations contained within the G-088 Get Away Special canister during the STS-007 mission (see EDSYN, STS-007 to review the other eight experiments). The objective of this experiment was to determine the effect of a low-gravity, vacuum environment on the performance of standard products/tools which might be used during future space missions.

A variety of items from companies such as (1) EDSYN, Inc., Van Nuys, California, (2) Hakko Metals, Co. Ltd., Osaka, Japan, and (3) ERSA, West Germany, were contained in eight storage drawers within the GAS canister. These items included soldering irons, assorted heaters, a temperature meter, electronic shears, a resoldering wick, vacuum pump plugs, a syringe, a check valve, an aluminum tip, a vacuum exhaust tool, a solder extractor heater, and a soldering tool holder. (See Reference (1), p. 15 for a detailed listing of the contents of each drawer. Model numbers are listed.)

All of the items were tested on Earth and in working order prior to the STS-007 flight. After placement of the items in the G-088 payload, the canister was evacuated to a 1 Torr vacuum level (a vacuum similar to that of the space environment).

Reportedly, all of the items were to be tested and evaluated after the mission. At the time Reference (1) was published, however, not all of the products had been tested. It was noted that all of the items which had been tested thus far "...function[ed] the same as before the flight with no degrada-

tion in performance." (1, p. 13)

Key Words: Technological Experiments, Soldering-Related Tools, Vacuum

Number of Samples: See Reference (1), p. 15.
Sample Materials: Please refer to the experiment summary above.
Container Materials: not applicable

Experiment/Material Applications:
"The tests were performed to determine if standard hand tools selected for space repair tasks would be able to withstand the rigors of outer space." (1, p. 4)

References/Applicable Publications:

(1) EDSYN NASA Payload No. 88, STS-7. Preliminary report, available from EDSYN Soldering Products, Inc., Van Nuys, California, 18 pp. (post-flight)

(2) "STS-7 Cargo Systems Manual: GAS," JSC-17645 Annex 7 Basic Version PCN-1, NASA JSC, April 1, 1983. (very short description; preflight)

(3) "STS-7 Getaway Specials," NASA News, NASA GSFC, May 1983.

(4) NASA STS-7 Seventh Space Shuttle Mission Press Kit, June 1983, p. 56. (preflight)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

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Affiliation(s): (1) During STS-017: Utah State University Faculty, Logan, Utah, Currently: ARME Enterprises, Hyrum, Utah; (2) Logan, Utah

Experiment Origin: USA
Mission: STS Launch #13, STS-017 (STS 41-G, Challenger)
Launch Date/Expt. Date: October 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: College Student Experiment
NASA Get Away Special (GAS) Canister G-518
Volume of Canister: 2.5 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-518: Utah State University, Logan, Utah
Processing Facility: Solder, heating elements, thin metal plates
Builder of Processing Facility: Unknown, possibly Utah State University, Logan, Utah

Experiment:
Solder-Flux Separation (Reflight of G-008 Soldering Experiment)

This experiment was one of four investigations housed within the G-518 Getaway Special Canister during STS-017. The three other experiments (of the four) were applicable to this data base (see Kitaura, STS-017 (Chapter 2); Thomas, S., STS-017 (Chapter 12); Walden, STS-017 (Chapter 15)).

Although information published prior to the STS-017 mission indicated that this experiment was a reflight of a soldering experiment by G. C. Alford (see Alford, STS-011, Get Away Special (GAS) canister G-008 (this chapter)), Alford verified that he was not the Principal Investigator of this investigation.

Few details describing the experimental setup and objectives could be located. Reference (1), which was released prior to the launch of STS-017, briefly described the expected experiment scenario. During the low gravity mission, "Solder is melted on two thin metal plates on opposite sides of the heating element...." (1, p. 2-7) The space-produced solder joints "...will be studied to determine the characteristics of the separation of solder and flux." (1, p. 2-7).

Only a very brief discussion of the experimental results could be located. Reportedly, the experiment proceeded as planned, but the flight samples appeared to have melted at a slower rate than

ground-based reference samples. It was suspected that this slower melting rate was due to either (1) a colder than expected GAS canister temperature or (2) low battery pack voltage.

No further discussion of the experiment could be located.

Key Words: Technological Experiments, Soldering, Flux, Melt and Solidification, Separation of Components, Surface Tension, Wetting, Solid/Liquid Interface, Material Strength, Thermal Environment More Extreme Than Predicted, Processing Difficulties, Battery Voltage Too Low

Number of Samples: unknown
Sample Materials: unknown, possibly samples of resin core and coreless solder
Container Material: The composition of the contacting metal plates was not identified.

Experiment/Material Applications:
See Alford, STS-004.

References/Applicable Publications:

(1) Cargo Systems Manual: GAS Annex for STS 41-G. JSC-17645 41-G, September 4, 1984. (short description; preflight)

(2) Space Shuttle Mission 41-G Press Kit, October 1984, pp. 24-25. (preflight)

(3) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(4) G-518 Payload Accommodations Requirements, NASA Goddard Space Flight Center, March 20, 1984.

(5) Press Release for G-518, Utah State University, Logan, Utah, 1984.

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Experiment Origin: USA
Mission: Skylab SL-3, Second Skylab Manned Mission
Launch Date/Expt. Date: August 1973 (month experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Skylab Student Project, High School Student Experiment, Skylab Manned Environment
Processing Facility: (1) Free cantilevered beam (spring loaded at the free end with different masses), (2) strain gauge to convert beam deflection into an electrical signal, (3) frequency counter, and (4) mass holder
Builder of Processing Facility: NASA Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Zero Gravity Mass Measurement (ED-74)

While the weight of an object cannot be directly measured in a near-weightless environment, the mass of an object can be measured under such conditions. During this Skylab-3 experiment, the ability to measure small masses in space was demonstrated.

The experimental apparatus consisted of an aluminum, cantilevered, flexible, spring beam (0.55 inch thick + or - 0.002 inch). The beam, which had a small cross section compared to its length, was fixed at one end to a frequency counter. The other end was free to oscillate.

During the experiment, one of five "test masses" was attached to the free end of the beam. The beam was then deflected from its resting position. The subsequent oscillation of the beam was sensed at the fixed end via strain gauges. These gauges, in turn, provided a signal to the frequency counter which had a visible readout of the vibration period (+ or - 0.001 second). A 16 mm camera documented the beam deflection, beam oscillation, and vibration period for all of the masses.

The value of each mass could be calculated from the period of oscillation. This period, T, is related to the unknown mass by the following equation:

T = 2*(pi) SQRT(M/K)

where M is the mass of the spring beam plus the unknown mass and K is the beam spring constant.

Prior to the flight, the apparatus was calibrated with known masses and a period vs. mass curve was constructed for flight experiment comparison. The flight data resulted in a similar curve which, when compared to the Earth-generated curve, plotted within the tolerances of the cantilever beam. It was reported that "There was a 3 to 4 percent difference between the ground-based data and the flight measured data but... [the difference] could have been attributed to an inexact knowledge of the beam's physical properties." (4, p. 78)

Key Words: Technological Experiments, Mass Measurement, Cantilevered Spring Beam, Mass-Spring Oscillation, Oscillation Frequency

Number of Samples: five applied masses
Sample Materials: The cantilevered spring beam was fabricated from an aluminum alloy.
Container Materials: not applicable

Experiment/Material Applications:
Skylab had two mass measurement devices onboard (for actual astronaut use) which used a spring-mass mechanical oscillator setup. However, "These two devices [did] not provide a clear demonstration of this mass measurement principle." (3, p. 6-54) This student experiment graphically demonstrated the principle for educational purposes.

This type of measurement device was only suited for the measurement of fixed mass quantities. It was unsuited for the measurement of a specific quantity required in a scientific experiment (e.g., 10 gm of sulfur). A variable quantity measurement could only be realized using an apparatus that simulated gravity, as would a centrifugal device. Such a (chemical balance) design was

detailed within the Principal Investigator's initial experiment proposal for Skylab.

Prior to the Skylab flight it was determined that such a chemical balance design was too difficult to implement prior to the mission. The design, which was later expanded in 1975, is detailed in Reference (5). The Principal Investigator surmised that such a device would make a useful addition to the space station measurement instruments.

References/Applicable Publications:

(1) "Mass Measurement (ED74)." In NASA MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, p. 12-83. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(3) "Experiment ED74 - Mass Measurement." In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 6-54 - 6-58. (post-flight)

(4) Skylab, Classroom in Space. Edited by Summerlin, L. B., NASA SP-401, 1977, pp. 77-80. (post-flight)

(5) Converse, V. W.: Final Report on Ed-74 Zero Gravity Mass Measurement. January 10, 1974. (post-flight)

(6) Mass Measurement (ED74). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-83 - 12-84.

(7) Input received from Principal Investigator V. W. Converse, August 1988.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 1
Launch Date/Expt. Date: December 1977
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 02-1 (resonant tube levitator with isothermal resistance-heated tube furnace)
Builder of Processing Facility: Levitator: Battelle-Institute, Frankfurt, Germany; Furnace: Unclear, possibly MBB/ERNO, Bremen, Germany

Experiment:
Acoustic Positioning

This TEXUS 1 experiment was the first in a series of investigations designed by Lierke to study the operation of an acoustic positioning device. The specific objective of the experiment was to determine if the device could suspend, melt, and solidify a molten metallic sample under low-gravity conditions. The device permitted (1) the containerless processing of a spherical material and (2) the study of the fluid physics of the sphere.

During the TEXUS 1 sounding rocket mission, a resonant tube leviator was contained in an isothermal, resistance-heated tube furnace (TEXUS Experiment Module TEM 02-1). The furnace was capable of moderate heat-up and cooling rates (less than or equal to 2 K/s). "The standing-wave resonance was temperature-compensated by an automatic gas injection system which readjusted the temperature-dependent wavelength change during heat-up and cooling by a concentration change of a two-component inert gas mixture (Krypton/Helium) with extremely different sound speed of the two components." (2, p. 1129)

A spherical sample (8 mm in diameter) of hypereutectic Ag-60 wt.% Sb was prepositioned within a wire cage. (The melt temperature of this alloy is 485 ^{o}C.) A cine camera was used during the rocket flight for sample observation.

It was reported that "Although the electronic monitor outputs indicated no malfunction during the flight, the cine recording showed that no stable positioning was achieved and that the sample touched the cage. Either the wave pattern was not stable or the gas which acted as an acoustic wave carrier was blown out of the furnace in the low pressure environment of the flight." (4, p. 360)

Very little additional information (related to the actual in-flight positioner/furnace operation) could be located at this time. References (1) and (2) discuss the theoretical background of this work.

<Note: E. G. Lierke was the Principal Investigator for the operation of the acoustic positioner; H. Ahlborn was the Principal Investigator for the Ag-Sb sample analysis. For details concerning the Ag-Sb sample analysis, see Ahlborn, TEXUS 1 (Chapter 6).>

Key Words: Technological Experiments, Melt and Solidification, Hypereutectics, Metals, Acoustic Positioning, Acoustic Levitation, Gas Injection, Resonant Frequency, Containerless Processing, Binary Systems, Spheres, Processing Difficulties

Number of Samples: one
Sample Materials: Ag-60 wt.% Sb; inert gas mixture: krypton/helium
(Ag*Sb*, Kr*He*)
Container Materials: not applicable

Experiment/Material Applications:
See Herlach, TEXUS 9 (Chapter 6).

References/Applicable Publications:
(1) Lierke, E. G., Grossbach, R., Flögel, K., and Clancy, P.: Acoustic Positioning for Space Processing of Materials Science Samples in Mirror Furnaces. Symposium in Industrial Activity in Space, Stressa, Italy, May 2-4, 1984, Proceedings, Paris, Eurospace, 1984, pp. 116-126. (post-flight)

(2) Lierke, E. G., Grossbach, R., Flögel, K., and Clancy, P.: Acoustic Positioning for Space Processing of Materials Science Samples in Mirror Furnaces. In Proceedings of 1983 Ultrasonics Symposium, October 31 - November 3, 1983, pp. 1129-1139. (post-flight; mentions TEXUS 9 proposal and includes theoretical discussion)

(3) Clancy, P. F., Lierke, E. G., Grossbach, R., and Heide, W. M.: Electrostatic and Acoustic Instrumentation for Material Science Processing in Space. Acta Astronautica, Vol. 7, 1980, pp. 877-891. (post-flight; discusses apparatus; no results)

(4) Acoustic Positioning of a Molten Alloy Sample. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 360-361. (post-flight)

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Co-Investigator(s): Unknown
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 9
Launch Date/Expt. Date: May 1984
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 02-2: monoellipsoidal mirror furnace (ELLI) combined with a resonant tube acoustic levitator.
Builder of Processing Facility: Levitator: Battelle-Institute, Frankfurt, Germany; Furnace: MBB/ERNO, Bremen, Germany

Experiment:
Acoustic Positioning

"Containerless processing under high purity conditions favours the extension of the undercooling range of metallic melts and can result in metastable crystalline or amorphous (glassy) phases with unique physical properties. Microgravity conditions facilitate the containerless processing of samples and prevent the sedimentation of denser components." (4, p. 362) <Note: See Herlach, TEXUS 9 (Chapter 6) for a more detailed discussion of the containerless processing goals.>

This TEXUS 9 experiment was the second in a series of investigations designed by Lierke to study the operation of an acoustic positioning device (see Lierke, TEXUS 1).

In preparation for the rocket flight, a resonant tube acoustic levitator was configured with the mirror furnace ELLI and placed in the TEXUS Experiment Module TEM 02-2). The levitator had been partially tested during parabolic aircraft flights (low-gravity periods of about 10 seconds) and was to be further tested during the TEXUS 9 sounding rocket mission (low-gravity period of 6 minutes). A Pd 77.5 - Cu 6 - Si 16.5 sample (8 mm in diameter, 2.84 gm) was prepositioned in a wire cage in the focus of the mirror furnace. <Note: It is not clear if the sample composition was in weight, volume, or atomic percentage.>

Reportedly, during the TEXUS 9 mission, "the experiment was performed in the temperature range 760 ^{o}C ([the sample] melting point) to 1000 ^{o}C with heating rates of 10 to 20 $^{o}C/s$. The sample was monitored with a CCD camera for automatic and telecommand control." (4, p. 362) <Note: The following is not clear: (1) if the sample was preheated prior to launch or (2) at what stage of the experiment the above temperature range was realized.>

Reportedly, "The positioning test was only marginally successful. The sample was effectively free floating while completely melted. However, it started to rotate and oscillate during the cooling phase and came into contact with the wire cage several times. Finally, it remained attached to the cage while still molten and solidified." (4, p. 362)

<Note: No other discussion concerning the experimental procedure or performance of the acoustic positioning device could be located at this time.>

<Note: E. G. Lierke was the Principal Investigator of the acoustic positioner/furnace operation. D. M. Herlach was the Principal Investigator of the sample analysis. For details concerning the sample analysis, see Herlach, TEXUS 9 (Chapter 6).>

Key Words: Technological Experiments, Melt and Solidification, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Containerless Processing, Ternary Systems, Alloys, Metals, Amorphous Materials, Sample Purity, Glasses, Glass Formation, Spheres, Drops, Drop Oscillation, Drop Rotation, Fluid Oscillation, Sample Rotation, Liquid Dynamic Response, Sedimentation, Undercooling, Processing Difficulties

Number of Samples: one
Sample Materials: 77.5% Pd-16.5% Si-6% Cu
(Pd*Si*Cu*)
Container Materials: not applicable

Experiment/Material Applications:
See Herlach, TEXUS 9 (Chapter 6).

References/Applicable Publications:
(1) Lierke, E. G., Grossbach, R., Flögel, K., and Clancy, P.: Acousitc Positioning for Space Processing of Materials Science Samples in Mirror Furnaces. In Symposium in Industrial Activity in Space, Stressa, Italy, May 2-4, 1984, Proceedings, Paris, Eurospace, 1984, pp. 116-126. (preflight)

(2) Lierke, E. G., Grossbach, R., Flögel, K., and Clancy, P.: Acoustic Positioning for Space Processing of Materials Science Samples in Mirror Furnaces. In Proceedings of 1983 Ultrasonics Symposium, October 31 - November 1-3, 1983, pp. 1129-1139. (preflight; mentions TEXUS 9 proposal; includes theoretical discussion)

(3) Clancy, P. F., Lierke, E. G., Grossbach, R., and Heide, W. M.: Electrostatic and Acoustic Instrumentation for Material Science Processing in Space. Acta Astronautica, Vol. 7, 1980, pp. 877-891. (preflight; discusses apparatus; no results)

(4) Acoustic Positioning. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 362-365. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 14a
Launch Date/Expt. Date: May 1986
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 02: monoellipsoidal mirror furnace (ELLI) combined with an acoustic positioning device. (TEM 02 was previously flown on TEXUS 9, but was now modified to improve the radial positioning force approximately 50% over TEXUS 9)
Builder of Processing Facility: Furnace: MBB/ERNO, Bremen, Germany <Note: It is not clear if the original builder of the levitator (Battelle-Institute, Frankfurt, Germany) also provided the newly modified acoustic positioner for this flight.>

Experiment:
<u>Acoustic Positioning</u>

This TEXUS 14a experiment was the third in a series of investigations designed by Lierke to study the low-gravity operation of an acoustic positioning device (see Lierke, TEXUS 1, TEXUS 9).

Details of the TEXUS 14a experimental setup and expected inflight experimental timeline were not discussed in the available publications. It appears that during the low-gravity experiment, a PdCuSi sample was to be positioned and heated using the TEXUS Experiment Module TEM 02-2. TEM 02-2 contained a monoellipsoidal mirror furnace with an acoustic positioning device.

Reportedly, because of an unexpected "wobbling motion" of the TEXUS rocket, uncontrollable accelerations were produced on the vehicle and the desired low-gravity level of 10^{-4} g was not attained. The experiment was reflown on TEXUS 14b (see Lierke, TEXUS 14b (this chapter)).

Documentation further detailing the results of this TEXUS 14a experiment does not appear to be available.

<Note: E. G. Lierke was the Principal Investigator of the acoustic positioner/furnace operation. D. M. Herlach was the Principal Investigator of the sample analysis (see Herlach, TEXUS 14a (Chapter 6)).>

Key Words: Technological Experiments, Melt and Solidification, Acoustic Positioning, Acoustic Levitation, Containerless Processing, Ternary Systems, Spheres, Acceleration Effects, Rocket Motion

Number of Samples: unknown, possibly one
Sample Materials: unknown, possibly PdCuSi alloy (Pd*Cu*Si*)
Container Materials: not applicable

Experiment/Material Applications:
See Lierke, TEXUS 9, **Experiment** section.

References/Applicable Publications:

(1) Experimentelle Nutzlast und Experimente TEXUS 14. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 53-55. (in German; post-flight)

(2) Lierke, E. G. and Grossbach, R.: Akusticshe Positionierung. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 90-93. (in German; post-flight)

(3) Experimentmodul TEM 02-2. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, p. 89. (in German; experiment module)

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Co-Investigator(s): Unknown
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 14b
Launch Date/Expt. Date: May 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 02-2: monoellipsoidal mirror furnace (ELLI) combined with an acoustic positioning device. (TEM 02 was previously flown on TEXUS 9, but was now modified to improve the acoustic radial positioning force by approximately 50% over TEXUS 9.)
Builder of Processing Facility: Furnace: MBB/ERNO, Bremen, Germany <Note: It is not clear if the original builder of the levitator (Battelle-Institute, Frankfurt, Germany) also provided the modified acoustic positioner.>

Experiment:
Acoustic Positioning

This TEXUS 14b experiment was the fourth in a series of investigations designed by Lierke to study the operation of an acoustic positioning device (see Lierke, TEXUS 1, TEXUS 9, TEXUS 14a). The device permitted the containerless processing of a spherical material. The specific objectives of the study were to (1) investigate the effect of large thermal gradients on an acoustic positioning system and (2) achieve the highest possible undercooling of a containerless sample.

The TEXUS Experiment Module TEM 02-2 was used for the experiment. The module contained a monoellipsoid reflecting furnace heated by a halogen lamp. A uniaxial acoustic positioning device equipped with the furnace was used to position a spherical PdCuSi sample. The sample position and lamp power could be adjusted by remote control during the mission.

Because of results from the earlier TEXUS 9 experiment (see Lierke, TEXUS 9) several improvements were made to the experimental apparatus. These improvements included:

(1) Increasing (by 50%) the radial positioning force of the acoustic positioner by (a) increasing the ultrasonic wave power and (b) optimizing the furnace tube diameter.

(2) Adding a second CCD camera to help assess the sample position and keep the sample within the furnace mirror focus.

(3) Using a newly developed damping mechanism which controlled the positioning force amplitude as a function of sample location.

(4) Isolating the inner part of the positioner from the mirror furnace volume by using (a) a bellows and (b) a high-purity protecting gas.

During the mission, a spherical Pd-Cu-Si alloy sample was suspended in the pressure junction point of a stationary ultrasound wave. The ultrasound wave was generated in a quartz tube connected to a sound transmitter.) The sample was observed with the two CCD cameras positioned at right angles to each other. (Reportedly, this camera configuration allowed exact monitoring of sample position.) Once positioned, the sample was melted and solidified. The furnace temperature was varied between room temperature and 900 ^{o}C at heating rates of 6 to 20 K/s and cooling rates of 4 to 8 K/s.

The furnace atmosphere was continually charged and purged with an inert gas to reduce the O_2 partial pressure as much as possible. This was required to retain sample purity. However, "Because of the piezo ceramic rings, contained in the built-in ultrasound converter, it was not possible to fully heat the interior part of the process chamber so that one must question the extremely low partial pressure [of O_2] (which was not measured). Besides, the flight sample... was not delivered melted in a quartz ampoule with a highly pure surface. Instead it had a visible impurity already during installation in the positioning unit which could not be removed [prior to flight] and which probably triggered germ formation in the undercooling experiment." (2, p. 91, translation)

It was reported that all components of the module operated perfectly during the experiment. While heating, the sample experienced radial oscillations which led to contact with the sample holder. However, as the temperature increased the oscillations disappeared and the specimen was calm while molten. The radial oscillations returned when the sample was cooled and the sample once again contacted the specimen holder.

<Note: E.G. Lierke was the Principal Investigator of the acoustic positioner/furnace operation. D. M. Herlach was the Principal Investigator of the sample analysis. For details concerning the sample analysis, see Herlach, TEXUS 14b (Chapter 6).>

Key Words: Technological Experiments, Melt and Solidification, Acoustic Positioning, Acoustic Levitation, Containerless Processing, Sample Purity, Undercooling, Ternary Systems, Eutectics, Alloys, Glasses, Drops, Drop Oscillation, Fluid Oscillation, Liquid Dynamic Response, Spheres, Thermal Gradient, Contamination Source, Impurities, Halogen Lamps

Number of Sample: one
Sample Materials: spherical PdCuSi alloy
(Pd*Cu*Si*)
Container Materials: not applicable

Experiment/Material Applications:
See Herlach, TEXUS 14b (Chapter 6).

References/Applicable Publications:

(1) Lierke, E. G., Grossbach, R., Flögel, K., and Clancy, P.: Acoustic Positioning for Space Processing of Materials Science Samples in Mirror Furnaces. Symposium in Industrial Activity in Space, Stressa, Italy, May 2-4, 1984, Proceedings, Paris, Eurospace, 1984, pp. 116-126. (preflight; TEXUS 1 results)

(2) Lierke, E. G. and Grossbach, R.: Akustische Positionierung. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 90-93. (in German; post-flight)

(3) Experimentmodul TEM 02-2. In BMFT TEXUS 13-16 Abschlussbericht 1988, pp. 90-93. (in German; post-flight)

(4) Lierke, E. G., Grossbach, R., Flögel, and Clancy, P.: Acoustic Positioning for Space Processing of Materials Science Samples in Mirror Furnace. In Proceedings of the 1983 Ultrasonic Symposium, October 31 - November 3, 1983, pp. 1129-1139. (preflight; theoretical discussion; TEXUS 1 results; TEXUS 9 proposal)

(5) Clancy, P. F., Lierke, E. G., Grossbach, R., and Heide, W. M.: Electrostatic and Acoustic Instrumentation for Material Science Processing in Space. Acta Astronautica, Vol. 7, 1980, pp. 877-891. (preflight; discusses experiment apparatus)

(6) Acoustic Positioning. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 366-369. (post-flight)

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Co-Investigator(s): Unknown
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 1
Launch Date/Expt Date: December 1977
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01: Isothermal Four Chamber Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Skin Technology

This TEXUS 1 experiment was the first of a series of investigations designed by Sprenger et al. to examine the feasibility of casting materials under low-gravity conditions using skin technology.

Low-gravity casting of materials using skin technology involves three major steps:

(1) On Earth, a part is cast or machined into its desired shape and a thin coating (skin) of an inert material (e.g., a ceramic) is applied.

(2) In the low-gravity environment, the part is melted and resolidified (e.g., directionally). In this environment an improved microstructure results because (a) sedimentation, convection and buoyancy effects are reduced and (b) the part can retain its complex casted shape.

(3) After low-gravity processing, the part is returned to Earth where the skin is either removed or left intact. (The skin is left intact if it can act as a protective layer.)

Before beginning low-gravity experiments in skin technology, certain questions and uncertainties had to be addressed:

(1) Prior to the experiment, it was generally accepted that single crystals, multivariant eutectics, and dispersion reinforced alloys would benefit from solidification under low-gravity conditions, although the degree of alloy refinement by space

processing had not been determined. Since these types of materials are candidates for turbine blade applications, they were considered important for study.

(2) Since Ni and Co based alloys (turbine blade materials) contain aggressive elements (Ti, Al, etc.), it was necessary to select a skin material which would remain stable during processing. Alumina was selected as the initial coating material, because (a) it has been employed extensively in industry and (b) it may be applied to the alloy by many different processes.

(3) The selection of alumina introduced additional uncertainties into the process. For instance, because of differences in thermal expansion between the alumina and sample material, stress might be created within the thin skin. Further, during melting, metallic alloys would exhibit positive volume increments which would introduce additional stress in the skin. The wetting characteristics of the melt with respect to the alumina would also introduce unknowns into the process.

Preliminary ground-based experiments employing simply shaped IN-100 sample materials (a Ni based turbine blade cast alloy, with plasma sprayed and detonation gun coating of alumina) were conducted in order to address some of the above mentioned uncertainties. It was found that:

(1) Although the skin retained its shape under the forces of gravity, the sample did not. However, the edges of the sample tended to add a stiffening effect.

(2) Skin destruction caused by (a) differences in thermal expansion properties between skin and sample materials and (b) volume expansion during melting were not observed.

(3) Thin skins (50 microns) tended to perform better than thick skins (200 microns) which tended to spall (flake off).

(4) The melt had excellent wetting behavior with the alumina skin.

The purpose of the TEXUS 1 experiment was to determine the differences between the 1-g processed samples and the low-gravity samples. For example, would be the deformations shown in the 1-g samples also be present in the low-g samples?

In preparation for the experiment, two samples (4 mm by 7 mm by 30 mm long) of IN-100 were each plasma spray coated with a 75 micron thick layer of alumina. In order to increase the wettability of the molten material to the skin, a 50 micron thick layer of NiAl was applied between the sample and alumina. The

samples were inserted in a Mo alloy (TZM) cartridge and placed within the gradient portion of the TEXUS Experiment Module TEM 01 isothermal furnace.

Previous testing of the furnace indicated that up to 2/3 of the samples could become molten using the maximum furnace temperature of 1600 ^{o}C. During the experiment, the samples were cooled with He gas directed along the outside of the Mo cartridge. Thermocouples could not be placed in the samples, so the actual thermal profiles were determined from calibration measurements.

Post-flight examination of the materials revealed that only one of the samples was melted. This anomaly was attributed to a "...non-symmetrical temperature distribution of the applied heating chamber." (2, p. 105)

The molten sample exhibited some deformation: an outward deformation of the middle section and an inward deformation of the section solidified last. It was reported that this deformation was due to a volume increase during melting which resulted in a pillow-shaped blowup of the sample. "At the beginning of resolidification this deformation of the skin remains fixed and can not be reversed within the solidified portion. Only after some time of solidification the decreasing volume of the sample leads to a compensating inward deformation." (2, p. 105)

It was reported that SEM microanalysis did not show evidence of segregation. Metallographic examination indicated that the unmelted portion had a recrystallized structure and the molten section had a cast structure.

Key Words: Technological Experiments, Melt and Solidification, Alloys, Directional Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Plasma Spray Coating, Wetting, Sample Deformation, Volume Expansion, Solid/Liquid Interface, Sample Microstructure, Turbine Blade Applications, Stress, Thermal Gradient, Thermal Expansion, Sedimentation, Quench Process, Buoyancy Effects Diminished, Convection, Asymmetric Temperature Field, Sample Not Processed as Planned

Number of Samples: two
Sample Materials: nickel-based alloy IN 100
(Ni*)
Container Materials: Skin material: plasma-sprayed coating of alumina (75 microns thick) with NiAl interlayer (50 microns thick); cartridge material: molybdenum alloy TZM
(Ni*Al*, Mo*)

Experiment/Material Applications:
The primary research application of skin technology is the production of turbine blades. Such production has a cost/benefits ratio which would be acceptable when confronted with the expense of low-gravity processing. Space-processed turbine blades could have in an increased temperature capability because of higher uniformity and lack of defects in the space processed structure. This microstructure improvement would be attributable to the lack of sedimentation and convection present during low-gravity processing. It was reported that these microstructure improvements would result in (1) doubling the blade's life expectancy, (2) reducing engine fuel consumption by 4%, and (3) increasing thrust by 10% (and thus increasing in payload potential by 20%).

(Other possible applications of skin technology can be located in Reference (5)).

IN-100 (a Ni-based superalloy) is a material which is used for turbine blade applications.

References/Applicable Publications:
(1) Sprenger, H. and Schweitzer, K.: Skin Casting Experiments in Rocket Flights. In Proceedings of the 5th ESA-PAC Symposium on European Rocket and Balloon Programmes & Related Research, Bournemouth (UK), April 14-18, 1980, ESA SP-152, June 1980, pp. 349-356.

(2) Sprenger, H., Erben, E., and Zeilinger, H.: Skin Technology. In ESA 3rd European Symposium on Material Science in Space, Grenoble, April 24-27, 1979, ESA SP-142, June 1979, pp. 101-108.

(3) Sprenger, H. and Erben, E.: Skin Technology-An Industrial Application of Space Processing. Acta Astronautica, Vol. 5, pp. 625-635. (Pre-TEXUS 1)

(4) Sprenger, H. and Schweitzer, K.,: Application of Skin Technology. Shuttle/Spacelab Utilization, Final Report, Project TEXUS II, pp. 10-26, 1978. (includes comparison to TEXUS I; post-flight)

(5) Sprenger, H. and Schweitzer, K.: TEXUS Experiments on Skin Technology. International Astronautical Federation, 32nd Congress, Rome, Italy, September 6-12, 1981, IAF-81-148, Preprint, 12 pp.

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(7) Sprenger, H. and Schweitzer, K.: Application of Skin Technology: TEXUS 2 Experiment. Bundesministerium fuer Forschung und Technologie, Report Number: BMFT-FB-W-81-028, December 1979, 63 pp. (in German; English summary; appears to include TEXUS 1 findings)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 2
Launch Date/Expt Date: November 1978
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: Isothermal Four Chamber Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Skin Technology

This TEXUS 2 experiment was the second in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-gravity conditions using skin technology (see Sprenger, TEXUS 1).

The specific objectives of the experiment were to (1) avoid sample deformation by volume expansion (such deformation had occurred in the TEXUS 1 skin technology sample), (2) demonstrate the ability to transform a sample prepared by powder-metallurgical techniques into a uniform cast structure under low-gravity conditions, and (3) improve (over the TEXUS 1 experiment) the temperature measurement of the samples.

Two different materials were selected for this experiment: (1) IN-100 cast Ni superalloy (the same alloy type as flown on TEXUS 1) and (2) a powder-metallurgically prepared IN-738 alloy with a Y_2O_3 particle dispersion. The samples (4 mm by 7 mm by 30 mm) were coated with a 50 micron thick skin of plasma-sprayed alumina. In order to compensate for volume expansion of the melt (approximately 3%), small holes were drilled through the skin into the specimen. It was hoped that these holes would prevent sample distortion. Each sample was fixed within a vacuum sealed Mo alloy (TMZ) cartridge by screw threads located at the top of the cartridge. The cartridges were contained in the gradient portion of the TEM 01-1 Experiment Module.

Three thermocouples provided temperature measurement. One thermocouple was located within an alumina spacer between the sample cartridges and the two other thermocouples were located in the

lower "cap" of each cartridge. The samples were to be melted and directionally solidified while under low-gravity conditions.

Post-flight analysis of the thermal data indicated that "The intended time-temperature profile of the TEXUS II experiment...was unfortunately not achieved due to a failure of two heating elements in the furnace which occurred at about 170 seconds after launch." (4, p. 15) Therefore, the samples did not achieve the desired temperature. However, more than 50% of each sample was melted. In addition, "...the [IN-100 alloy] sample had become unscrewed from the cartridge before or during launch of the TEXUS rocket so that it was free during the flight." (4, p. 16) No such problem was reported for the IN-738 sample.

Metallurgical examination of the IN-100 material revealed the presence of a Ni-Mo eutectic at one end of the sample. This presence indicated contact between the melt and the Mo alloy cartridge. "The heavy deformation of the edges in the upper section of the specimen could have been caused by the formation of... Ni-Mo eutectic alloy by sucking off the liquid alloy through the bore. The deformation was more severe than [that observed] in 1-g experiments... and may... have been intensified by [the] inner surface tension of the hole which was obviously closed by a metallic film. The possibility that the perhaps non-solidified specimen collided with the cartridge walls during gravitational re-entry of the rocket seems unlikely." (4, p. 17) Below this eutectic section, the sample contained a dendritic structure of a few mm in length. Following this section was a mushy zone which indicated this portion of the sample had existed between the liquid and solid states.

Similar results were found with the IN-738 sample. The microstructure present in over half of the sample indicated only partial melting. In addition, residual gas cavities, present because of the powder-metallurgic preparation, existed within the sample. These gas inclusions expanded during melting and led to a severe deformation of the sample. Metallographic studies also indicated that the Y_2O_3 particles had agglomerated at the grain boundaries. This agglomeration may be attributed to (1) the solidification interface pushing the particles through the melt and/or (2) the induced motion of the melt by the gas inclusions (although this factor cannot be confirmed).

Although the heaters failed, some positive observations could be made. Despite the deformation of the IN-100 sample, the volume expansion holes proved effective in avoiding the problems associated with volume expansion of the melt. (The 1-g experiments also confirmed this finding.) It was reported that the good wettability of the skin by the melt prevented discharge of the melt through the hole in the skin. It also appeared that good wet-

tability is an important factor in skin technology becasue it prevents detachment of the melt and skin.

Key Words: Technological Experiments, Melt and Solidification, Directional Solidification, Mushy Zone, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Plasma Spray Coating, Dispersion Alloys, Powder Metallurgy, Particle Dispersion, Particle Agglomeration, Particle Motion, Sample Microstructure, Wetting, Wetting of Container, Surface Tension, Capillary Flow, Sample Deformation, Volume Expansion, Cavity, Solid/Liquid Interface, Turbine Blade Applications, Thermal Gradient, Thermal Expansion, Eutectics, Dendritic Structure, Grain Boundaries, Inclusions, Crucible Effects, Sample Detachment from Crucible, Vacuum, Furnace Malfunction, Processing Difficulties, Rocket Motion, Vehicle Re-Entry Forces/Vibration

Number of Samples: two
Sample Materials: Sample #1: nickel based alloy IN-100; sample #2: nickel based alloy IN-738 containing approximately 0.1 micron Y_2O_3 dispersed particles
(Ni*, Y*O*)
Container Materials: Skin material: plasma-sprayed alumina, Al_2O_3; cartridge material: molybdenum alloy TMZ
(Al*O*, Mo*)

Experiment/Material Applications:
The specific reason why the IN 738 alloy (with a Y_2O_3 dispersion) was selected was not detailed in available publications. However, it is believed that the IN 738 alloy is used for turbine blade applications. Dispersion strengthened alloys are also used for turbine blades.

See also Sprenger, TEXUS 1.

References/Applicable Publications:
(1) Sprenger, H. and Schweitzer, K.: Skin Casting Experiments in Rocket Flights. In Proceedings of the 5th ESA-PAC Symposium on European Rocket and Balloon Programmes & Related Research, Bournemouth (UK), April 14-18, 1980, ESA SP-152, June 1980, pp. 349-356.

(2) Sprenger, H., Erben, E., and Zeilinger, H.: Skin Technology. In ESA 3rd European Symposium on Material Science in Space, Grenoble, April 24-27, 1979, ESA SP-142, June 1979, pp. 101-108.

(3) Sprenger, H. and Erben, E.: Skin Technology-An Industrial Application of Space Processing. Acta Astronautica, Vol. 5, pp. 625-635. (pre-TEXUS 1)

(4) Sprenger, H. and Schweitzer, K.: Applications of Skin Technology. Shuttle/Spacelab Utilization Final Report Project Texus II 1978, pp. 11-26. (German publication)

(5) Sprenger, H. and Scweitzer, K.: TEXUS Experiments on Skin Technology. International Astronautical Federation 32nd Congress, Rome Italy, September 6-12, 1981, IAF-81-148, Preprint, 12 pp.

(6) Sprenger, H. and Schweitzer, K.: Application of Skin Technology: TEXUS 2 Experiment. Bundesministerium fuer Forschung und Technologie Report Number: BMFT-FB-W-81-028, December 1979, 63 pp. (in German; English summary)

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(8) Sprenger, H. J.: Directional Solidification of Metals and Alloys. Appl. microgravity Tech. 1, 1987, pp. 30-36. (post-flight)

(9) Skin Technology. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 340-341. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 3
Launch Date/Expt Date: April 1980
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2
Builder of Processing Facility: Unknown, possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
<u>Skin Technology</u>

This TEXUS 3 experiment was the third in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-gravity conditions using skin technology (see Sprenger, TEXUS 1, TEXUS 2).

During the previous two TEXUS experiments in this investigative series the furnace temperature was not high enough to completely melt the samples. Therefore, it was proposed that a low-melting, aluminum-based alloy be employed for the TEXUS 3 experiment. An $AlMg_3$ alloy was selected not only for its melt temperature, but because a thin skin could be applied to its surface by either an anodic or chemical oxidation process.

Prior to the mission, two samples were prepared. For the first sample, a thin skin was applied to the $AlMg_3$ sample by a process of electrolytic oxidation in ammonium tartrate. (The available thickness of the skin, which was far less than 1 micron, created difficulties during ground experiments.) For the second sample, a (<1 micron thick) skin (produced by natural oxidation) was allowed to form on the $AlMg_3$. Other changes/improvements in the experimental setup included (1) the omission of the Mo alloy cartridges which, on previous low-gravity experiments, contained the coated samples and (2) the placement of thermocouples within the sample in axially drilled bores. The samples were processed using the gradient portion of the TEM 01 experimental module.

Post-flight analysis of the processed materials revealed severe deformation of the sample although the thin skin prevented any spillage of the melt. It was concluded that the deformation was caused by an unintended time-temperature profile which resulted

in the introduction of the cooling gas into the furnace chamber while the samples were still molten. "The temperature differences in relation to the ground experiments... [was] obviously due to the fact that the heat flow from furnace to sample was reduced due to the low gas pressure in the furnace during the rocket flight." (1, p. 8)

Reportedly, because of a rocket de-spin failure, the intended low-gravity level of this mission was never achieved during the TEXUS 3 flight. However, no publications could be located which discussed the effects of this higher gravity level on the results of the skin technology experiment.

Key Words: Technological Experiments, Melt and Solidification, Directional Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Oxidation, Alloys, Binary Systems, Wetting, Sample Deformation, Solid/Liquid Interface, Turbine Blade Applications, Thermal Gradient, Quench Process, Gas Pressure, Acceleration Effects, Rocket Motion, Rocket Despin Failure, Hardware Malfunction

Number of Samples: two
Sample Materials: aluminum-magnesium alloy Al-Mg_3
(Al*Mg*)
Container Materials: skin material produced by electrolytic oxidation in ammonium tartrate or natural oxidation
(N*H*, O*)

Experiment/Material Applications:
See Sprenger, TEXUS 1.

The $AlMg_3$ alloy was selected for this experiment because of its low melting point. Further, a thin skin could be applied to the alloy by either an anodic or chemical oxidation process.

References/Applicable Publications:
(1) Sprenger, H. and Schweitzer, K.: TEXUS-Experiments on Skin Technology. XXXII Congress of International Astronautical Federation, Rome, Italy, pp. 1-11, September 6-12, 1981. (post-flight)

(2) Skin Technology. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February, 1991, p. 342. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 3b
Launch Date/Expt Date: April 1981
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experimental Module TEM 01: (Reconfigured after TEXUS 3)
Builder of Processing Facility: Unknown, possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Skin Technology

This TEXUS 3b experiment was the fourth in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-gravity conditions using skin technology (see Sprenger, TEXUS 1, TEXUS 2, TEXUS 3). Originally, the experiment was to be a repeat of the investigation flown during the TEXUS 3 mission. However, the TEXUS 3 mission demonstrated that the employed Al alloy samples could not be successfully processed using external cooling. Therefore, an entirely new experimental design employing active pedestal cooling was chosen for the TEXUS 3b flight.

The TEM 01 furnace module of the TEXUS rocket was reconfigured for TEXUS 3b such that a single $AlMg_3$ sample was attached by a 5 mm screw to a He cooled support structure. It was reported that the sample could be directionally solidified within about three minutes using this configuration. A 2 mm diameter bore was drilled axially through the sample and four thermocouples (one of which was used for temperature control) were inserted in the bore.

A new skin coating process was used for the sample. The material was dipped into SnO_2, thus coating the $AlMg_3$ with a 0.3 micron thick layer of the ceramic. This process coated (1) the outside of the sample and (2) the inner thermocouple bore. Finally, "The front face was... [ground] so that the 'container' was open at the side where melting would begin in order to allow the melt to expand into free space and in this way to avoid corresponding forces on the side faces of the sample." (1, p. 10)

The time-temperature profile of the flight experiment was not reported in the available literature.

Post-flight examination of the sample revealed severe deformation, indicating (1) that residual forces acted on the sample and (2) the sample coating thickness was not sufficient to withstand these forces. The sample exhibited regions of crumpling and inward deformation indicative of fluid motions within the melt. There was also a twisting of the open front face of the sample (approximately 25^{o} clockwise). An oxide layer was observed covering the open face which acted as a skin to stabilize this area of the sample. In the section which solidified last, the SnO_2 skin was corrugated with a wavelength of 1.1 mm. This corrugation may have been caused by instabilities produced by volume expansion of the alloy during melting. However, this corrugation behavior had not been observed during ground experiments.

It was also reported that there was an increase in sample volume. This increase was attributed to voids caused by volume shrinkage during solidification in the last solidified section of the sample. The presence of these voids indicated "...the applied experimental design was not effective in compensating the volume alteration...." (1, p. 11) It was also noted that the inner skin within the cavity was very stable. This tends to confirm a main advantage of skin technology: during melting and solidification, inner cavities will remain stable.

Key Words: Technological Experiments, Melt and Solidification, Directional Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Alloys, Binary Systems, Ceramics, Wetting, Free Surface, Sample Deformation, Volume Expansion, Cavity, Voids, Sample Shrinkage, Solid/Liquid Interface, Turbine Blade Applications

Number of Samples: one
Sample Materials: aluminum-magnesium alloy, $AlMg_3$
(Al*Mg*)
Container Materials: skin material: dip-coated tin oxide, SnO_2
(Sn*O*)

Experiment/Material Applications:
See Sprenger, TEXUS 1; Sprenger, TEXUS 3.

A SnO_2 dip-coated skin was used because (1) it can be applied to all types of alloys and (2) it coats both the outer surface and the inner cavities. Ground-based experiments indicated that the skin illustrated good stability against metallostatic pressures and did not react with the molten alloy.

References/Applicable Publications:

(1) Sprenger, H. and Schweitzer, K.: Texus Experiments on Skin Technology. 32nd International Astronautical Federation Congress, Rome, Italy, September 6-12, 1981, IAF-81-148, Preprint, 12 pp.

(2) Skin Technology. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February, 1991, p. 342. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 4
Launch Date/Expt Date: May 1981
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01: (reconfigured after TEXUS 3)
Builder of Processing Facility: Unknown, possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Skin Technology

This TEXUS 4 experiment was the fifth in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-g conditions using skin technology (see Sprenger, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b). The experiment was performed to supplement the earlier TEXUS 3b investigation.

Examination of the TEXUS 3b sample after low-gravity processing revealed that the specimen had been severely deformed. This deformation was attributed to the thinness of the skin (approximately 0.3 microns). Therefore, the objective of this TEXUS 4 experiment was to process a sample using a thicker coating.

It was reported that all TEXUS 4 experiment parameters (including sample composition ($AlMg_3$)), were the same as those of the TEXUS 3b experiment except that the TEXUS 4 sample skin thickness was 2 microns (see Reference (1)). <Note: It was reported in a later paper (see Reference (2)) that the skin thickness of the TEXUS 4 sample was 3 microns.> No discussion of the time-temperature profile for the TEXUS 3b or TEXUS 4 experiments was provided.

Post-flight examination of the TEXUS 4 sample revealed that the specimen was severely deformed. The deformation was not, however, as great as that of the TEXUS 3b sample, which illustrated the contribution of skin thickness to shape stability. The open front face of the TEXUS 4 sample exhibited a twisting of 10° clockwise (compared to 25° anticlockwise for the TEXUS 3b sample).

It was reported that "Forces de to interfacial tension were... responsible for the deformation of the TEXUS IV sample.... Because of insufficient thickness the skin was not rigid enough to retain the original shape of the sample...." (2, p. 89) An earlier publication, which discussed the TEXUS 3b and TEXUS 4 results, reported that "The deformation as such is not... [explained] by spheroidization according to surface tension since the original length of the sample is fully restored. Also crumpling and regions of inward deformation indicate that fortuitous motions of the melt must have caused the observed disfiguring of the specimens." (1, p. 10)

All other observations of the TEXUS 4 sample were the same as those reported for the TEXUS 3b specimen (see Sprenger, TEXUS 3b).

When the results from TEXUS 1 and TEXUS 2 were compared with those from TEXUS 3, TEXUS 3b, and TEXUS 4, the following conclusions were reported:

(1) The most intensive forces acting against shape stability are (a) volume expansion during melting and (b) shrinkage during solidification.

(2) Extremely thin skins (less that 10 microns) may have been influenced by additional forces (other than those mentioned above) during low-gravity processing. For instance, gas flow from other furnace chambers may have passed into the processing chamber, thus deforming the skin.

(3) "The elimination of free surface forces by covering the alloy with a good wetting skin seems to be effective as no tendency of spheroidization of the samples could be observed in all experiments." (1, p. 11)

(4) Additional stabilization may be provided by the insertion of cavities into the specimen.

Key Words: Technological Experiments, Melt and Solidification, Directional Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Alloys, Binary Systems, Wetting, Surface Tension, Free Surface, Free Surface Elimination, Interfacial Tension, Sample Deformation, Volume Expansion, Sample Shrinkage, Gas Injection, Solid/Liquid Interface, Turbine Blade Applications

Number of Samples: one
Sample Materials: aluminum-magnesium alloy, $AlMg_3$ (Al*Mg*)
Container Materials: skin material: dip-coated tin oxide, SnO_2 (Sn*O*)

Experiment/Material Applications:
See Sprenger, TEXUS 3b.

References/Applicable Publications:

(1) Sprenger, H. and Schweitzer, K.: Texus Experiments on Skin Technology. 32nd International Astronautical Federation Congress, Rome, Italy, September 6-12, 1981, IAF-81-148, Preprint, 12 pp.

(2) Sprenger, H.: Skin Castings of Alloys and Composites - Results of SL-1 and TEXUS Experiments. In Proceedings of 5th European Symposium on Material Sciences Under Microgravity, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 87-94. (post-flight)

(3) Skin Technology. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 344-345. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 5
Launch Date/Expt Date: April 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: Isothermal Four Chamber Furnace
Builder of Processing Facility: Unknown, possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Skin Technology

This TEXUS 5 experiment was the sixth in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-gravity conditions using skin technology (see Sprenger, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 4).

Although Reference (1) indicated that the experiment was performed during the TEXUS 5 mission, no discussion of the experimental objectives, setup, or results could be located.

Key Words: Technological Experiments, Melt and Solidification, Directional Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Solid/Liquid Interface, Turbine Blade Applications

Number of Samples: unknown
Sample Materials: unknown
Container Materials: unknown

Experiment/Material Applications:
See Sprenger, TEXUS 1.

References/Applicable Publications:

(1) Walter, H. U.: Results of Materials-Science Experiments with Sounding Rockets. ESA Journal, Vol. 7, No. 3, 1983, pp. 235-256. (post-flight)

(2) Sprenger, H: Skin Casting of Alloys and Composites Results of SL-1 and Texus Experiments. In ESA 5th European Symposium on Material Sciences Under Microgravity, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 87-94. (post-flight; this reference may be applicable; it provides a general discussion of the skin technology experiments performed during the TEXUS missions)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 7
Launch Date/Expt Date: May 1983
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2: Isothermal Four Chamber Furnace
Builder of Processing Facility: Unknown, possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Skin Technology

This TEXUS 7 experiment was the seventh in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-gravity conditions using skin technology (see Sprenger, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 4, TEXUS 5).

Although Reference (1) indicated that the experiment was performed during the TEXUS 7 mission, no discussion of the experimental objectives, setup, or results could be located.

Key Words: Technological Experiments, Melt and Solidification, Directional Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Solid/Liquid Interface

Number of Samples: unknown
Sample Materials: unknown
Container Materials: unknown

Experiment/Material Applications:
See Sprenger, TEXUS 1.

References/Applicable Publications:

(1) Communication with the DFVLR, Federal Republic of Germany.

(2) Sprenger, H: Skin Casting of Alloys and Composites Results of SL-1 and Texus Experiments. In ESA 5th European Symposium on Material Sciences Under Microgravity, Schloss Elmau, November 5-7, 1984, ESA Publication SP-222, pp. 87-94. (post-flight; This reference may be applicable; it provides a general discussion of the skin technology experiments performed during the TEXUS missions.)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility
Processing Facility: Isothermal Heating Facility (IHF) Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Skin Technology (1ES303)

This Spacelab 1 experiment was the eighth in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-gravity conditions using skin technology (see Sprenger, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 4, TEXUS 5, TEXUS 7).

The previous experiments in this investigative series were conducted during sounding rocket flights throughout the TEXUS program. The results from the TEXUS experiments, as well as ground-based studies, led to the conclusions that the main contributions to sample skin deformation were (1) volume change during melting and resolidification and (2) interfacial forces between the melt and applied skin. These early experiments also indicated that these detrimental forces could be countered by the use of (1) a sufficiently stiff (thick) skin and (2) a non-wetting expansion reservoir (which compensated for sample volume change).

The objectives of this Spacelab 1 experiment were to (1) confirm the low-gravity shape stability of a liquid metal contained by a ceramic skin and (2) determine the microstructural differences between a 1-g and low-gravity processed monovariant eutectic alloy.

The eutectic sample selected for the mission consisted of a gamma/gamma(superprime) - alpha, Ni-Al-Mo alloy. <Note: It appears that prior to the flight, an aluminum oxide coating was to be applied to the sample. However, no publication, published after the flight of the STS-009 mission, could be located which confirmed that the sample was coated with the aluminum oxide as planned.>

Reportedly, the sample was to be directionally solidified in the Spacelab Isothermal Heating Facility (IHF). However, "...this experiment could not be performed in SL-1 [Spacelab 1] due to breakdown of the IHF power supply." (1, p. 90)

No further information concerning the objectives, procedures, or results of this experiment could be located.

Key Words: Technological Experiments, Melt and Solidification, Directional Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Alloys, Ceramics, Ternary Systems, Eutectics, Wetting, Interfacial Tension, Interface Phenomena, Solid/Liquid Interface, Sample Deformation, Volume Change, Volume Compensation, Turbine Blade Applications, Hardware Malfunction, Sample Not Processed As Planned

Number of Samples: one
Sample Materials: eutectic alloy of gamma/gamma(superprime)-alpha nickel-aluminum-molybdenum
(Ni*Al*Mo*)
Container Materials: skin material: unknown, possibly alumina (Al*O*)

Experiment/Material Applications:
See Sprenger, TEXUS 1.

The specific reason why the skin material was chosen was not presented in available publications. It was stated that the sample material, (the Ni-Al-Mo alloy) was "...of technical interest...." (1, p. 90)

See also, Sprenger, Spacelab D1, WL-IHF-03 (this chapter).

References/Applicable Publications:
(1) Sprenger, H.: Skin Casting Of Alloys and Composites: Results Of Spacelab (SL)-1 and TEXUS Experiments. In ESA 5th European Symposium on Material Sciences Under Microgravity. Results Of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 87-94. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. Teledyne Brown Engineering Publication (acceleration measurements on Spacelab 1; post-flight)

(3) Sprenger, H. J.: Directional Solidification of Metals and Alloys. Appl. Microgravity Tech., 1, 1987, pp. 30-36. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 9
Launch Date/Expt Date: May 1984
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: Isothermal Furnace
Builder of Processing Facility: Unknown, possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Skin Technology

This TEXUS 9 experiment was the ninth in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-gravity conditions using skin technology (see Sprenger, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 4, TEXUS 5, TEXUS 7, Spacelab 1).

The specific objective of the experiment was to control the shape of a molten alloy sample under low-gravity conditions. The objective was to be accomplished by (1) using a thin coating of ceramic material which is poorly wetted by the molten alloy, (2) using a hole in the top of the skin which compensates for volume expansion during melting, and (3) preventing oxidation of the alloy free surface.

During the TEXUS 9 flight, a rectangular silver sample, coated with a 20 micron thick skin of TiO_2/Ni, was directionally solidified. The specimen had "...a cavity whose volume is in the order of the volume increase [of the sample material] during melting. Assuming that the melt does not leave the skin containment[,] the developing free surface should move back during solidification according to the wetting behavior of the system." (2, p. 88) It was believed that if solid oxides were not on the free surface of the silver melt, "...this design should work." (2, p. 88).

Post-flight analysis of the sample indicated that there was no deformation of the skin. However, the large wetting angle between the skin and alloy and the large capillary forces were responsible for the detachment of a portion of the melt from the

edges and corners of the skin. It was concluded that an optimum design of a skin coating should be (1) well wetted by the melt in those areas where shape conservation of the sample is required and (2) poorly wetted by the melt in those areas used for volume compensation.

Key Words: Technological Experiments, Melt and Solidification, Directional Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Ceramics, Alloys, Wetting, Non-Wetting of Container, Contact Angle, Surface Tension, Free Surface, Capillary Forces, Solid/Liquid Interface, Oxidation, Thermal Gradient, Volume Retention, Sample Detachment from Crucible, Turbine Blade Applications

Number of Samples: one
Sample Materials: silver alloy
(Ag*)
Container Materials: skin material: titanium oxide, TiO_2, with nickel
(Ti*O*, Ni*)

Experiment/Material Applications:
See Sprenger, TEXUS 1.

The reasons why silver was selected as the sample material or TiO_2 with Ni was selected as the skin coating were not detailed in available publications.

References/Applicable Publications:
(1) Sprenger, H. J.: Directional Solidification of Metals and Alloys by Means of Skin Technology. Appl. Microgravity Tech., 1, 1987, pp. 20-36. (post-flight)

(2) Sprenger, H.: Skin Casting of Alloys and Composites Results of SL-1 and Texus Experiments. In ESA 5th European Symposium on Material Sciences Under Microgravity, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 87-94. (This reference may be applicable, mission numbers are unspecified.)

(3) Skin Technology. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 346-347. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 11
Launch Date/Expt Date: April 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: Isothermal Four-Chamber Furnace Module with sample pedestal
Builder of Processing Facility: Unknown, possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Protective Film Engineering: Skin Technology

During the protective film engineering process, a skin-coated alloy is refined by melting and resolidifying a cast material. Throughout the processing, the alloy retains its original shape.

This TEXUS 11 experiment was the tenth in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-gravity conditions using skin technology (see Sprenger, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 4, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 9). The experiment was the first of a two part investigation; the second part was performed during the TEXUS 12 mission (see Sprenger, TEXUS 12).

Earlier TEXUS studies in this investigative series had illustrated the importance of (1) providing for the volume change of sample materials during the melt and resolidification process and (2) counteracting interfacial forces which can cause sample shape deviation. It was found that these issues can be addressed by (1) the use of non-wetting expansion reservoir (which compensates for volume changes) and (2) the use of a skin of sufficient stiffness (thickness) which can be wetted by the melt.

Thus, the joint objectives of the TEXUS 11 and TEXUS 12 experiments were:

(1) "- conservation of the sample shape by means of a suitable volume compensation system and of a well wetted skin;

(2) "- study of the behaviour of model dispersion alloys during melting and solidification. In particular the influence of the wetting behavior of the Cu-matrix [melt] on both the Mo

[protective] skin and the dispersed particles (Al_2O_3 or Mo) was to be studied." (2, p. 348)

During ground-based and earlier TEXUS experiments (in this investigative series) it was determined that particles which are wetted poorly by the melt (e.g. Al_2O_3 particles in Cu melt) tend to be "...pressed out of the melt under 1g, [and] under low gravity they may be slightly shifted or twisted by the solidification front...." (1, p. 16, translation) However, particles which are appropriately wetted (e.g. Mo particles in Cu melt) tend to be well distributed throughout the matrix.

In an effort to investigate the solidification of particles with different wetting behaviors, a Cu-Al_2O_3 particle dispersion sample was processed during this experiment and a Cu-Mo particle sample was processed during the TEXUS 12 experiment.

Prior to the TEXUS 11 flight, a Cu sample containing a 0.1% dispersion of Al_2O_3 particles was prepared by powder-metallurgical techniques. The sample was plasma coated with Mo (which is wetted by molten copper). A reservoir for volume expansion was included. The reservoir had been coated with Al_2O_3 which is not wetted by the Cu melt. <Note: The exact location of the reservoir was unclear to the editors.>

The TEXUS 11 sample was processed in one of the four chambers of the TEXUS Experiment Module TEM 01-1. Before launch, the furnace was preheated to 900^oC. Once in flight, the sample was melted at a temperature of 1070^oC. The sample was directionally solidified using a sample pedestal which acted as a cooling base.

Post-flight analysis of the sample revealed that the melted and re-solidified portion of the material contained holes that ranged in size from about 20 microns to 2 mm in diameter. The holes which were under 100 microns were round while those larger than 100 microns "...have an irregular shape in the lower portion of the sample, with sometimes relatively thin walls between cavities." (1, p. 17, translation) The alumina particles had agglomerated in the holes and at the free surfaces which were both formed during the volume expansion. It was determined that the holes were created by CO formation. These types of holes were absent during ground-based experiments since the bubbles could rise through the melt. It was reported that "Due to the motions induced in the melt, no conclusion could be drawn with respect to the experiment objectives." (2, p. 348)

No other information concerning this experiment could be located at this time.

Key Words: Technological Experiments, Melt and Solidification, Directional Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Plasma Spray Coating, Dispersion Alloys, Powder Metallurgy, Binary Systems, Particle Distribution, Particle Dispersion, Particle Agglomeration, Particle Motion, Wetting, Solid/Liquid Interface, Interfacial Tension, Thermal Gradient, Volume Expansion, Volume Compensation, Sample Deformation, Non-wetting of Container, Gas Formation, Bubbles, Absence of Buoyancy Forces (Detrimental), Turbine Blade Applications

Number of Samples: one
Sample Materials: copper powder with 0.1% Al_2O_3 particles (Cu*Al*O)
Container Materials: skin material: plasma-sprayed molybdenum (Mo*)

Experiment/Material Applications:
See Sprenger, TEXUS 1.

See the above **experiment** summary for a discussion of sample material selection.

References/Applicable Publications:

(1) Sprenger, H.: Protective Film Engineering. In TEXUS 11/12 Abschulussbericht 1985. (in German; post-flight)

(2) Skin Technology. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 348-349. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 12
Launch Date/Expt Date: May 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1 (upgraded from TEM 01-1)
Builder of Processing Facility: Unknown, possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Protective Film Engineering: Skin Technology

This TEXUS 12 experiment was the eleventh in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-gravity conditions using skin technology (see Sprenger, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 4, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 9, TEXUS 11). The experiment was also the second of a two-part investigation; the first part took place during the TEXUS 11 flight (see Sprenger, TEXUS 11). The joint objectives of the TEXUS 11 and TEXUS 12 experiments were:

(1) "- conservation of the sample shape by means of a suitable volume compensation system and of a well wetted skin;

(2) "- study of the behaviour of model dispersion alloys during melting and solidification. In particular the influence of the wetting behavior of the Cu-matrix [melt] on both the Mo [protective] skin and the dispersed particles (Al_2O_3 or Mo) was to be studied." (2, p. 348)

The TEXUS 12 sample was a powder-metallurgically prepared copper material which (1) had 0.1% dispersion of Mo particles and (2) was plasma coated with Mo film. All other reported experimental parameters were the same as the TEXUS 11 investigation.

Post-flight analysis of the low-gravity sample indicated that the Mo particles were uniformly distributed over the entire melted region. Unlike the TEXUS 11 sample, the specimen was practically free of holes. When examined under higher magnification, it was observed that "...the particles [had] layered onto each other within the melt to form a rigid network." (1, p. 17,

translation) Reportedly, "...the particle network formation ability depends on their volume fraction in the molten matrix. The effect of an increased motion or extenal forces (e.g. floatation of bubbles on Earth...) has already been observed and showed that particle networks become solid only at higher volume fraction of particles." (2, p. 350)

Other conclusions were presented:

"The mesh width of the network also... [appeared]... to depend on the motion in the melt (for constant volume percentage). The results can be interpreted that the TEXUS 12 sample... [had]... a clearly smaller mesh width of the network (about 100 microns) than that of the ground samples (about 200 microns), and as the extreme case, we [had] formation of a single mesh, i.e. the complete emptying of the sample of particles.

"Regarding the behavior of the protective film we [found] that the improved wetting from the use of Ar/H_2 atmosphere on the ground, was not observed. <Note: This was the only reference to the Ar/H_2 atmosphere.>

"The form stability of the melts within the protective film (characterized in TEXUS 9 by complete separation of the edges in the molten state from the inside of the protective film) was significantly increased in TEXUS 12. The conclusion from TEXUS 9 that the wetting behavior was the decisive criterion for form stability of film-coated grains, was reinforced. The differently long release of the melts from the edges of the protective film [indicated] that the degree of wetting (determined e.g. by the contact angle in the Young relation) is not an absolute constant quantity over a larger surface." (1, pp. 18-19, translation)

Key Words: Technological Experiments, Melt and Solidification, Directional Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Plasma Spray Coating, Binary Systems, Powder Metallurgy, Dispersion Alloys, Homogeneous Dispersion, Particle Distribution, Particle Dispersion, Surface Tension, Wetting, Contact Angle, Solid/Liquid Interface, Thermal Gradient, Volume Retention, Volume Compensation, Sample Detachment from Crucible, Interface Physics, Turbine Blade Applications

Number of Samples: one
Sample Materials: copper powder with molybdenum particles (Cu*Mo*)
Container Materials: skin material: plasma-sprayed molybdenum (Mo*)

Experiment/Material Applications:
See Sprenger, TEXUS 1 and Sprenger, TEXUS 11 **Experiment** section.

References/Applicable Publications:
(1) Sprenger, H.: Protective Film Engineering. In TEXUS 11/12 Abschulussbericht 1985. (in German; post-flight)

(2) Skin Technology. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 348-350. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility with Gradient Device (IHF/G)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Skin Technology/Eutectic Solidification (WL-IHF-03)

The Spacelab D1 experiment was the twelfth in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-gravity conditions using skin technology (see Sprenger, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 4, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 9, TEXUS 11, TEXUS 12). The investigation was the second of two experiments specifically designed to study the low-gravity directional solidification of a coated eutectic alloy (see Sprenger, Spacelab 1).

Objectives of this Spacelab experiment included (1) confirming the use of skin technology for long-term shape stability and for the prevention of cavities within the melt and (2) investigating the convective-free solidification of metallic alloys.

The sample material selected for the investigation was a gamma/gamma(superprime)-alpha, Ni-Al-Mo alloy. It was hoped that directional solidification of this alloy would result in a regular arrangement of Mo fibers (with a high aspect ratio) contained within a Ni/Ni_3Al (gamma/gamma(superprime)) matrix. The material is highly sensitive to various solidification parameters (e.g., growth rate, thermal gradient, compositional fluctuations) and, therefore, was expected to provide information concerning convective flow patterns immediately ahead of the solidification interface.

The sample (7 mm diameter, 150 mm long) was plasma-spray coated with an 80 micron thick skin composed of ZrO_2-7.5% Y_2O_3. The sample was cylindrical with the exception of the last section

which had a flat longitudinal face. For safety reasons, the coated sample was placed within an alumina tube. A volume compensation bore was drilled into the end of the sample exhibiting the flat face. Four thermocouples were included within the sample to define the thermal parameters.

The Spacelab D1 Isothermal Heating Facility 'with gradient device' (IHF/G) was used to process the eutectic alloy. In order to investigate the transition from aligned to cellular growth, the sample was subjected to four translation rates from 0.1 to 0.5 mm/min during the mission. The last 45 mm of the sample was quenched (complete details concerning the time-temperature profile of the sample can be found in Reference (4)).

Post-flight analysis of the space-processed sample indicated that there was no deformation of the outer skin or the sample. The shape and stability of the end with the flat face were excellent. No pores or holes could be detected throughout the length of the sample indicating that the skin was wetted by the melt. Microstructural examination of the sample indicated that there was no detachment of the melt from the skin and, thus, a suppression of surface-driven convection was realized.

These results illustrated that processing within a wetted skin was possible and that this technique is (1) important in regards to the suppression of surface-driven convection under low-gravity conditions and (2) a simple way to compensate for volume change during melting and solidification without the use of an external device.

Microstructural examination of the flight sample showed five distinct regions, separated according to the expected four translation rates and an additional, unexpected translation rate. The first region (approximately 0.3 mm of the sample) was solidified at an unintended rate of about 0.025 mm/min due to "...non-equal pulling velocity of the furnace." (4, p. 346) The microstructure exhibited a coarse eutectic with periodic thickening of Mo fibers. The first 1 mm of the second region (rate = 0.145 mm/min) contained an irregularly shaped carbide phase with a composition of Ni_6Mo_6C. The remainder of the second region (approximately 10 mm) consisted of parallel Mo fibers and parallel blades of the carbide phase within a Ni/Al matrix. The carbide phase was not observed in 1-g processed samples except "...as a blocky phase in the periphery region where the liquid/solid phase boundary is curved...." (6, p. 352) The first 4-5 mm of the next region (rate = 0.259 mm) also consisted of the parallel eutectic growth with carbide blades. However, the interfiber distance was decreased. After 4-5 mm, the parallel structure broke down and became cellular. This transition occurred without change in the growth rate or thermal gradient.

The next two regions (rate = 0.37 mm/min and 0.48 mm/min) consisted of a continuance of the cellular structure. However, the cell structure was much larger and the cell length was much longer than that seen in similarly processed samples under 1-g conditions.

Analysis of the above results and comparison of low-gravity and 1-g processed samples indicated that the main difference in microstructures between the materials was due to the presence of the carbide phase in the space sample. The first 1 mm section of the space sample did not contain a carbide phase indicating "...that during the early stage of solidification a boundary layer [had] formed which continuously was enriched with carbon. The formation of the carbide band occurred as the solubility limit for carbon was surpassed." (6, p. 353) During processing under 1-g conditions, the transport of the carbon was increased due to convection. This increase in transport velocity resulted in less carbon within the boundary layer and thus the carbides were not formed. The formation of the Mo_6Ni_6C carbide also acted to shift the Ni and Mo content at the solidification interface.

In the 1-g processed samples, the transition from aligned to cellular growth occurs at a translation rate of about 0.3 mm/min. When processed under low-gravity conditions, this transition occurred at a rate of 0.259 mm/min. It was assumed that the difference in transisiton rate was due to the "...compositional shift along the monovariant trough caused by the continuous precipitation of... carbides from the melt ahead of the moving interface." (6, p. 353)

A complete discussion of the results and a description of a possible model of the convection at the interface can be found in Reference (6).

Key Words: Technological Experiments, Melt and Solidification, Directional Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Plasma Spray Coating, Alloys, Eutectics, Fiber Eutectics, Fibers, Ternary Systems, Wetting, Wetting of Container, Free Surface Elimination, Solid/Liquid Interface, Interface Physics, Solidification Front Physics, Boundary Layer, Surface Tension-Driven Convection, Marangoni Convection, Marangoni Convection Diminished, Volume Retention, Volume Compensation, Sample Deformation, Sample Microstructure, Porosity, Cavity, Cellular Morphology, Precipitation, Growth Rate, Translation Rate, Pulling Rate, Thermal Gradient, Quench Process, Turbine Blade Applications, Hardware Malfunction

Number of Samples: one
Sample Materials: eutectic alloy of gamma/gamma(superprime)-alpha nickel-aluminum-molybdenum
(Ni*Al*Mo*)
Container Materials: skin material: plasma-sprayed ZrO_2-7.5% Y_2O_3; cartridge material: Al_2O_3, alumina
(Zr*O*Y*O*, Al*O*)

Experiment/Material Applications:
See Sprenger, TEXUS 1 (Skin Technology)

Earlier experiments, performed on the ground and during the TEXUS program, indicated that thin (30 to 100 micron) coatings were sufficient to retain the shape of a cast material during melting and resolidification under low-gravity conditions. If the skin had good wetting properties with respect to the melt, then the formation of pores between the melt and skin should be suppressed. (The formation of pores resulted in localized fluid flow caused by Marangoni convection and thus had a detrimental effect on the solidification of the sample.)

It is believed that the study of solidification of metallic alloys under low-gravity conditions will lead to (1) improved understanding of solidification processes on Earth and (2) production of materials with improved properties in space. Directionally solidified, eutectic alloys (in which aligned fibers or lamellae are embedded within a matrix) are of particular interest.

Earlier studies had shown that low-gravity processing of eutectics can lead to (1) an improved microstructure and (2) an increase in the fiber density. However, the results from low-gravity processing have been limited to a small number of alloy systems. Therefore, it was decided to study a eutectic alloy, (gamma/gamma(superprime)-alpha) Ni-Al-Mo, which has important high temperature applications.

The specific reason why the ZrO_2-7.5% Y_2O_3 skin was employed was not detailed in available publications.

References/Applicable Publications:
(1) Sprenger, H.: Skin Technology-Directional Solidification of Multiphase Alloys. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney (Germany), August 27-29, 1986, pp. 36-37. (post-flight)

(2) Sprenger, H.: Skin Technology. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, p. 147. (preflight)

(3) Sprenger, H.: Stutzhauttechnologie. Natur wissenschaften, 73.Jahrgang Heft 7, July 1986, pp. 390-395. (in German; post-flight)

(4) Sprenger, H. J.: Skin Technology-Directional Solidification of Multiphase Alloys. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 342-349. (post-flight)

(5) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986. (post-flight; acceleration measurements on D1)

(6) Sprenger, H. J.: Directional Solidification of a Eutectic Alloy Results of the D-1 Experiment. In Proceedings of the Sixth European Symposium on Material Sciences under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 349-354. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility (IHF) Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Skin Casting on Grey Cast Iron (WL-IHF-07)

This Spacelab D1 experiment was the third in a series of investigations designed by Sprenger and/or Luyendijk et al. to study the low-gravity directional solidification of cast iron (see Luyendijk, TEXUS 6, Spacelab 1 (Chapter 14)). The experiment was also the thirteenth in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-gravity conditions using skin technology (see Sprenger, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 4, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 9, TEXUS 11, TEXUS 12, Spacelab D1 experiment WL-IHF-03).

The objectives of this Spacelab D1 experiment were to (1) examine the graphite growth in a eutectic, grey cast iron sample (with low sulfur content) during directional solidification, (2) determine the diffusion of the sulfur in liquid cast iron, and (3) examine the directional solidification of a material using skin technology. (The investigation was performed in parallel with D1 experiment WL-IHF-03 (see Sprenger, Spacelab D1, experiment number: WL-IHF-03 (this chapter)).)

The employed cylindrical cast iron sample (6.5 x 149 mm) was comprised of two separate regions. Region 1 (had been directionally solidified prior to the flight and contained 4.34 wt.% C, 1.04 wt.% Si, and 0.008 wt.% S. The region had a small neck section which was used during the experiment to investigate the effect of a sudden change in diameter on the resulting microstructure. Region 2 contained 4.61 wt% C, 0.31 wt% Si, and 0.005 wt% S. A

small plug of iron-sulfide phase material (0.15 wt.% S) was located at one end of region 2 and used for the diffusion of sulfur study.

The sample contained four thermocouples: three within region 1 and one within region 2. The entire sample was plasma spray coated with an alumina skin (Al_2O_3) approximately 80 microns thick. Reportedly, the wetting angle between the cast iron and alumina skin was greater than 90 ^{o}.

During the Spacelab D1 flight, the sample was processed in the Isothermal Heating Facility (IHF). First, the furnace was evacuated and heated to 1350 ^oC. Next, the sample was melted directionally from region 2 to region 1 (in order to avoid separation of the liquid column) and then directionally solidified, from region 1 to region 2. During the directional solidification process, two different furnace translation rates were to be employed: 0.1 mm/min for 140 min and 0.3 mm/min for 35 min.

It was reported in Reference (2) (a document published prior to the Spacelab D1 flight) that region 2 would be quenched in order to freeze the sulfur concentration. However, no references published after the D1 mission could be located which confirmed this quenching procedure.

Post-flight analysis of the time-temperature profile revealed "...temperature fluctuations over distinct periods of time. They are not caused by fluctuations in furnace temperature." (3, p. 350) The largest fluctuations were measured at the thermocouples placed immediately before and immediately after the neck region of the sample. "That suggests that the cooling of the gradient device and/or the moving of the furnace was not stable. Whatever the reason may be, temperature fluctuations are more or less fatal to growing eutectics unidirectionally." (3, p. 350)

There also appeared to be a problem with the translating mechanism of the furnace. Under nominal conditions, the furnace moves over the sample to its position for the start of solidification. "At the end of this stroke the furnace goes into some blocking device with metallic springs. When, however, at the planned moment of time the motordrive is switch to the reverse direction [to begin directional solidification], it appears that for a period of about 20 minutes the gradient device does not move. There is a dead stroke due to the blocking system of the furnace." (3, p. 350)

Metallographic examination of region 1 indicated the presence of alternating bands of fine and coarse graphite of varying widths. These bands corresponded well with the temperature fluctuations.

Within the slow translation rate region of the sample, three actual growth rates were determined from the position of the fine/coarse graphite bands: 0.128, 0.221, and 0.231 mm/min. The actual growth rate of 1-g processed sample was 0.124 mm/min. The reason for this difference in growth rate was unclear to the investigators. The actual growth rate of the high translation rate region of the sample was estimated to be 0.42 mm/min, which was higher than the 0.338 mm/min reported for the 1-g processed sample.

A graph of the low-gravity interlamellar spacing versus growth rate was compared to similar data obtained from 1-g experiments. It appears that the lamellar spacing was not significantly different in the low-gravity and 1-g processed materials (at least for growth rates in the range of 2.0×10^{-4} to 6.0×10^{-4} cm/sec).

X-ray fluorescence methods were used to determine the distribution of sulfur in the longitudinal section of region 2. This examination revealed a build up of sulfur on the B side (side away from the iron-sulfide plug) of region 2. This behavior was not observed in the 1-g processed sample and was most likely due to the directional melting of the flight sample: "When side A [side containing the iron-sulfide plug] of region 2 starts melting, side B [had] been superheated already, so a distinct temperature gradient is across region 2 in that period of the experiment.

"As mentioned above the sulphur in the...[plug]...is present as a separate phase in the solid material. During melting droplets of iron-sulphide will be formed, that have a distinct lifetime before they have soluted in the warm liquid. In that period of time, due to the temperature gradient, the small droplets or at least a great part of it [sic] move to the hotter side of region 2 by a Marangoni effect. If the temperatures of side A and B have been the same, apparently no or no strong convection occurs." (3, p. 354)

Post-flight examination of the sample's ceramic skin indicated high stability during the experiment. There was also no reaction between the sample and alumina skin. The only damage to the specimen was at the top of the skin which was broken. A few liquid droplets were pressed out due to expansion of the melt. An examination of the liquid meniscus at the top of the sample indicated a non-wetting of the skin by the melt. Therefore, "...at the thermocouple grooves the existence of a free surface during the experiment should not be excluded." (3, p. 351)

Key Words: Technological Experiments, Melt and Solidification, Directional Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Plasma Spray Coating, Ceramics, Eutectics, Diffusion, Wetting, Contact Angle, Free Surface, Free Surface Elimination, Surface Tension, Meniscus Shape, Sample Necking, Volume Expansion, Non-wetting of Container, Solid/Liquid Interface, Solidification Front Physics, Thermal Gradient, Superheating, Translation Rate, Growth Rate, Multiphase Media, Lamellar Structure, Sample Microstructure, Liquid Columns, Drops, Drop Formation, Marangoni Movement of Droplets, Dissolution, Quench Process, Hardware Malfunction

Number of Samples: one
Sample Materials: eutectic grey iron, Fe-4.3 wt.% C-0.5 wt.% Si-0.005 wt.% S
(Fe*C*Si*S*)
Container Materials: skin material: plasma-sprayed alumina, Al_2O_3; cartridge materials: unknown
(Al*O*)

Experiment/Material Applications:
See Sprenger, TEXUS 1 (Skin Technology)

Grey cast iron is a material which has many technological applications. The structure of the material consists of graphite flakes within an iron matrix. The size and distribution of these flakes determine the mechanical properties of the cast iron and are governed by solidification parameters such as cooling rate, impurity level (e.g., sulfur), and convection within the melt. The individual and/or combined influence of these parameters is not yet well defined. Earth studies have not been able to resolve the contributions of these parameters because sulfur causes significant convection in the liquid which tends to overrule the influence of factors such as growth rate and thermal gradient.

References/Applicable Publications:

(1) Malinowski, M., Nieswaag, H., and Sprenger, H.: Skin Casting of Grey Cast Iron. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, pp. 110-111. (post-flight)

(2) Sprenger, H. and Nieswagg, H.: Skin Casting of Grey Cast Iron. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 147-149. (preflight)

(3) Nieswaag, H. and Malinowska, M.: Skin Casting of Grey Cast Iron. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 349-355. (post-flight)

(4) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1. Norderney, Germany, August 27-29, 1986. (post-flight; acceleration measurements on D1)

(5) Sprenger, H. J.: Directional Solidification of Metals and Alloys. Appl. Microgravity Tech. 1, 1987, pp. 30-36. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 15
Launch Date/Expt. Date: May 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1
Builder of Processing Facility: Unknown, possibly the Swedish Space Corporation, Solna, Sweden

Experiment:
Dispersion Alloys

Very little information concerning this experiment could be located. However, it appears that this TEXUS 15 sounding rocket experiment was the fourteenth in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-g conditions using skin technology (see Sprenger, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 4, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 9, TEXUS 11, TEXUS 12, 2 experiments on Spacelab D1).

Although the objective of the experiment was not specifically stated, it appears that the overall goal of the investigation was to study the stability of model dispersion alloys (each coated with a protective skin) during melting and resolidification. The sample material(s) selected for the investigation were not reported and the experimental setup was not described in any detail.

It was reported that shortly after the successful launch of the TEXUS 15 rocket, data and television transmitters experienced a partial failure. It was discovered that a lateral burnthrough of the second stage of the rocket had occurred, and the stage in turn, had collided with the prematurely separated payload. The upper part of the payload, including the TEM 01-1 module parachuted to the Earth undamaged.

Documentation, which details any results of the experiment does not appear to be available.

Key Words: Technological Experiments, Melt and Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Dispersion Alloys, Solid/Liquid Interface, Rocket Failure, Payload Survivability

Number of Samples: unknown
Sample Materials: unknown
Container Materials: unknown

Experiment/Material Applications:
See Sprenger, TEXUS 1 and TEXUS 11 **Experiment** section.

References/Applicable Publications:

(1) Experimentelle Nutzlast und Experimente TEXUS 15. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 107-108. (in German; post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 16
Launch Date/Expt. Date: November 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1
Builder of Processing Facility: Unknown, possibly the Swedish Space Corporation, Solna, Sweden

Experiment:
Dispersion Alloys

Very little information concerning this experiment could be located. However, it appears that this TEXUS 16 sounding rocket experiment was the fifteenth in a series of investigations designed by Sprenger et al. to study the feasibility of casting materials under low-g conditions using skin technology (see Sprenger, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 4, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 9, TEXUS 11, TEXUS 12, 2 experiments on Spacelab D1, TEXUS 15).

Although the objective of the experiment was not specifically stated, it appears that the overall goal was to study the stability of model dispersion alloys (each coated with a protective skin) during melting and resolidification. The sample material(s) selected for the investigation were not reported and the experimental setup was not described in any detail.

It was reported that shortly after the successful launch of TEXUS 16, fuel in the second stage of the rocket did not ignite as planned. After the apogee was reached and the rocket began to fall, the yo-yo despin system was deployed as programmed. Due to the unexpected excess rocket mass however, there was an incomplete reduction of rocket spin. Subsequently, the payload separated from the second stage. Unfortunately the accompanying parachute was not released. An unbraked impact of the payload resulted in the destruction of all experiment modules including the TEM 01-1 module.

Documentation, which details any results of the experiment does not appear to be available.

Key Words: Technological Experiments, Melt and Solidification, Skin Technology, Skin Casting, Coated Surfaces, Thin Films, Dispersion Alloys, Solid/Liquid Interface, Rocket Failure, Payload Survivability

Number of Samples: unknown
Sample Materials: unknown
Container Materials: unknown

Experiment/Material Applications:
See Sprenger, TEXUS 1 and TEXUS 11 **Experiment** section

References/Applicable Publications:
(1) Die Kampagne TEXUS 16. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 109-111. (in German; post-flight)

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Co-Investigator(s): Unknown
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 3
Launch Date/Expt. Date: April 1980
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Unknown, possibly: monoellipsoid mirror furnace
Builder of Processing Facility: Unknown, possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Halogen Lamp Performance

This TEXUS 3 experiment was the first in a series of investigations designed by Eyer et al. to evaluate the low-gravity performance of a halogen lamp.

A description of the specific experimental objectives and equipment setup of the payload could not be located.

Reportedly, due to a rocket despin failure, TEXUS 3 did not achieve the desired low-gravity level. The experiment was reflown on TEXUS 3b (see Eyer, TEXUS 3b).

Documentation detailing any results of this TEXUS 3 experiment does not appear to be available.

Key Words: Technological Experiments, Halogen Lamps, Acceleration Effects, Rocket Despin Failure

Number of Samples: unknown
Sample Materials: unknown
Container Materials: unknown

Experiment/Material Applications:
Unknown

References/Applicable Publications:

(1) Eyer, A., et al.: Preparation of Crystal Growth Experiments in Spacelab- Si (FSLP), CdTe (D1) and ZnS (D1). BMFT-FB-W-84-045, 147 pp. (in German)

(2) Greger, G.: TEXUS and MIKROBA and Their Effectiveness and Experimental Results. Presented at In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP). (identifies rocket failure)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 3b
Launch Date/Expt. Date: April 1981
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Monoellipsoid mirror furnace
Builder of Processing Facility: Unknown, possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Halogen Lamp Performance

This TEXUS 3b experiment was the second in a series of investigations designed by Eyer et al. to evaluate the low-gravity performance of a halogen lamp (see Eyer, TEXUS 3).

A description of the specific experimental objectives and equipment setup of the payload could not be located. Documentation detailing any results of this TEXUS 3b experiment does not appear to be available.

Key Words: Technological Experiments, Halogen Lamps

Number of Samples: unknown
Sample Materials: unknown
Container Materials: unknown

Experiment/Material Applications:
Unknown

References/Applicable Publications:
No publications could be located which discuss the TEXUS 3b experiment objectives, setup, or results.

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Co-Investigator(s): Unknown
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Experiment Origin: Japan
Mission: TT-500A 12 (Materials Processing Flight #5)
Launch Date/Expt. Date: January 1983
Launched From: Takesaki Launch Site in Tanegashima Island (Tanegashima Space Center)
Payload Type: Sounding Rocket Experiment
Processing Facility: Halogen Lamp
Builder of Processing Facility: Unknown

Experiment:
Halogen Lamp

This TT-500A sounding rocket experiment was designed to examine the performance of a halogen lamp under low-gravity conditions. The specific objective of the experiment was to determine if "...the halogenous cycle occurred in the microgravity environment." (1, p. 2) <Note: Reference (2) provides a brief discussion of the halogen cycle.>

No discussion of the experimental setup could be located at this time.

A brief discussion of the experimental results reported that "...the halogenous cycle seems to have occurred in the microgravity environment, because the halogenous tungsten did not make the black phenomena on the walles [sic] of the halogenous lamps." (1, p. 2) Reference (1) contains a plot of the halogen lamp current versus time. It appears that the current was constant (with the exception of a few spikes) at about 3.5 amps from approximately 105 seconds to approximately 460 seconds after launch.

No further information concerning this experiment could be located at this time.

Key Words: Technological Expeirments, Halogen Lamps, Halogen Cycle

Number of Samples: not applicable
Sample Materials: halogen lamp with tungsten filament (W*)
Container Materials: not applicable

Experiment/Material Applications:
"Halogenous lamps are planned to be used as heat sources of an image furnace." (1, p. 2)

References/Applicable Publications:
(1) Kajiwara, K., Matsuda, T., Shibato, Y., Masuda, T., and Akimoto, T.: Results of Japanese Space Processing Experiments in the TT-500A Rocket. 34th International Astronautical Federation, International Astronautical Congress, Budapest, Hungary, October 10-15, 1983, IAF Paper #83-157, 9 pp. (very short summary; post-flight)

(2) Ara, T.: Japan Microgravity Project. In 2nd Joint Japan-Germany-ESA Symposium on Microgravity Research, Tokyo, March 25-26, 1985, pp. 57-60. (post-flight)

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Experiment Origin: USA
Mission: STS Launch #10, STS-011 (STS 41-B, Challenger)
Launch Date/Expt. Date: February 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) Canister G-051
  Volume of Canister: 5 cubic feet
  Location of Canister: STS payload Bay
Primary Developer/Sponsor of G-051: GTE Laboratories, Inc., Waltham, Massachusetts
Processing Facility: Three metal halide arc lamps
Builder of Processing Facility: GTE Laboratories, Inc., Waltham, Massachusetts

Experiment:
Study of Convection-Free Metal Halide Lamps

"When operated on Earth, gravity induces circulation of the hot gases in... [high intensity metal halide] arc lamps. That circulation, or convection, affects the electrical and light-producing properties of the arc. These effects, mixed with others, are difficult to separate in ground-based experiments. The observations made while gravity...[is] "switched off" provide verification of theories of arc behavior, clarify the roles of convection versus other processes in the arc, and may lead to potential product improvements that result from altering the influence of convection.

"In metal halide lamps an arc is established in an inner capsule, or arc tube, which has metal electrodes protruding through its ends to pass electrical current through the gas inside. The gas is mostly mercury vapor with small amounts of sodium and scandium added to improve the color of the radiated light. During normal operation convection results in segregation of the various species, an effect which impacts the color and efficiency of the light source." (1, p. 17-18)

During this Space Shuttle experiment, the first detailed study of gravity-free arc operation was realized. The three lamps tested were of the metal halide type: a mercury lamp with sodium and scandium additives to whiten the otherwise bluish color of Hg lamps. All three were 175 W Metalarc[R] brand lamps.

During the experiment, the lamps were turned on and allowed to warm up and stabilize (typically 5-8 minutes). They remained on for a total of 30 minutes. Reportedly, "The arcs were photographed to record their general structure and, by means of three bandpass filters, to record the emission from mercury, sodium and scandium. In addition, a record was made of arc current, arc voltage, relative light intensity and arc tube wall temperature." (1, p. 18) Other experimental observations included qualitative records of more subtle phenomena such as cataphoresis of additive species.

The observed space arc operation was compared to terrestrial arc operation (and thus data have been obtained with gravity-induced convection absent as well as present). Reportedly, "Evaluation of digital film data [from the experiment] shows that the 175 watt Metalarc® [brand]...lamp has a significant increase in light output when convection is removed in the gravity free environment.... This increase in efficacy is due to a more uniform temperature and radiating species distribution. Operation under DC power reveals sizable cataphoretic effects that are being studied further." (1, p. 24)

Other conclusions included:

(1) the short time periods associated with previous free-fall reduced-gravity experiments were insufficient for examining steady-state properties of additive lamps,

(2) convection and cataphoresis are of comparable influence on the operation of a metal halide arc,

(3) when the plasma is operated at a frequency of 60 Hz in space, the additives are distributed uniformly along the arc axis,

(4) when the plasma is operated at very low frequencies, cataphoresis is sufficient to effectively remove additives from the plasma column (i.e., when operated with dc power, metal halide lamps effectively revert to simple mercury arc lamps).

<Note: Not all of the publications listed in the Applicable References section below were available to aid in the writing of this experiment summary.>

Key Words: Technological Experiments, Metal Halide Arc Lamps, Arc Behavior, Electric Field, Electrodes, Cataphoresis, Buoyancy-Driven Convection, Segregation, Gaseous Convection, Buoyancy Effects Diminished, Thermal Distribution

Number of Samples: three
Sample Materials: metal halide arc lamps with surrounding gaseous environment consisting of 1 atmosphere of dry nitrogen
Container Materials: not applicable

Experiment/Material Applications:
The results from this study provide valuable insights for lamp design as well as for analyses of fundamental aspects of lamp arc operation. (Please refer to the Experiment section (above).)

References/Applicable Publications:
(1) Bellows, A. H. and Feuersanger, A. E.: Arc Discharge Convection Studies: A Space Shuttle Experiment. In NASA Goddard Space Flight Center's 1984 Get Away Special Experimenter's Symposium, NASA CP-2324, August 1-2, 1984, pp. 17-24. (post-flight)

(2) Cargo Systems Manual: GAS Annex for STS-11, JSC-17645 Annex STS-11, December 2, 1983, pp. 2-3 - 2-4. (short description; preflight)

(3) Bellows, A. H., Feuersanger, A. E., Rogoff, G. L., and Rothwell, H. L.: HID [High Intensity Discharge] Convection Studies: A Space Shuttle Experiment. Illuminating Engineering Society Meeting (1984).

(4) Bellows, A. H., Feuersanger, A. E., Rogoff, G. L., and Rothwell, H. L.: Convection and Additive Segregation in High-Pressure Lamp Arcs: Early Results from a Space Shuttle Experiment. Gaseous Electronics Conf. (1984), Bull. Amer. Phys. Soc., Vol. 30, p. 141 (1985). (post-flight)

(5) Rogoff, G. L., Feuersanger, A. E., Bellows, A. H., and Rothwell, H. L.: Convection and Additive Segregation in Metal-Halide Lamp Arcs: Results from a Space Shuttle Experiment. Symposium on Science and Technology of High Temperature Light Sources, Electrochemical Society Meeting, Toronto, May 1985, Extended Abstracts, Vol. 85-1, Abstract No. 385, p. 551. (post-flight)

(6) Bellows, A. H., Feuersanger, A. E., Rogoff, G. L., and Rothwell, H. L.: HID Convection Studies: A Space Shuttle Experiment. Lighting, Design and Applications. (to be published) <Note: The current status of this document is unclear at this time.>

(7) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(8) STS-11 Getaway Special Payload Descriptions. NASA News, NASA GSFC, 1984.

(9) Getaway Special (GAS) Payloads (STS-11). In Goddard Space Flight Center's Engineering Newsletter, Vol. 2, No. 3, April 1984, p. 9. (very short description)

(10) Input received from Experiment Investigator, September 1989 and July 1993.

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #13, STS-017 (STS 41-G, Challenger)
Launch Date/Expt. Date: October 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) Canister G-013
Volume of Canister: 5 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-013: Kayser-Threde, Munich/Dornier System, Friedrichschafen, Germany
Processing Facility: Ellipsoidal mirror and halogen lamp
Builder of Processing Facility: Kayser-Threde, Munich, Germany

Experiment:
Halogen Lamp Experiment (HALEX)

Mirror heating facilities, which are used for crystal growth and other material science experiments, are usually configured with one or two halogen lamps. The lamps provide the heat for the facility; the mirrors focus the lamp radiation onto the melting specimen.

The major objectives of this STS-017 Get Away Special experiment were to (1) examine the low-gravity operation of a halogen lamp during an extended period (approximately 60 hours) and (2) illustrate the lamp's low-gravity capabilities under conditions similar to the lamp operating in a furnace configuration.

It was expected that the experiment would illustrate:

"*[The] Radiative behavior of a Halogen lamp during long-term operation in space

"*[The] Tungsten deposition inside [the] bulb if not retransported on to [the] filament

"*[The] Performance of the Halogen Cycle" (2, p. 142)

The experiment hardware was configured with several items including: (1) a sealed ellipsoidal mirror shell with vacuum port, (2) a HALEX 45 W lamp filled with 10 cm^3 xenon and admix-

tures at a pressure of approximately 4.5 bars, (3) two photocells for light detection, (4) two heat pipes for heat transfer from the lamp base to an intermediate plate, (5) temperature sensors at the photocells, lamp base, heat pipes, and intermediate plate, (6) eight 27 Vdc batteries, (7) a data acquisition system, and (8) two redundant tape recorders. In preparation for the experiment, a photocell (instead of a sample) was inserted within the focus of the reflected light.

It appears that the experiment was activated fairly early in the mission (approximately 34 hours after launch). Soon after the system "warm-up" was complete (the warm-up took 3 minutes) the operating setting was initialized.

Approximately 16 hours after the activation of the experiment, the shuttle SIR-B antenna was deployed. This antenna unexpectedly cast a shadow on the GAS payload and inhibited heat rejection from the canister. <Note: It was not clear why shadowing of the payload by the SIR-B antenna caused the cited inhibited heat rejection.> Thus, the payload experienced a significant temperature increase. As a result of the increased temperature, the lamp switched off automatically 56 hours after payload activation, because the upper temperature limit of the heat pipes had been exceeded. (During these 56 hours "long term lamp operation" was realized.) One-half hour later, the lamp automatically switched back on after the temperature had reached a tolerable level. Approximately 2 hours later, the payload was automatically switched off "due to a low voltage power cut-off." (57.9 hours was approximately 10% of the expected life span for the envisioned space flight lamp.)

Evaluation of the payload performance indicated that:
"* Lamp voltage was constant over the whole experiment period

"* Lamp current was constant

"* Resistance of [the] lamp filament did not change (<0, 1%)

"* Photo signals were constant with respect to the radiation input

"* Lamp base temperature showed that [the] lamp bulb temperature was as expected

"* Heat pipe temperatures showed [the] proper function...

"* No detectable disturbances of the Halogen cycle [had occurred] (i.e. no deposit of Tungsten on the bulb)

"* Surface characteristics of the filament [were] as expected (microscopic inspection)" (2, pp. 148-149)

It was, therefore, concluded that:

"* The absence of convection (under microgravity) inside the lamp bulb results in a reduction of convective heat transfer from 5% to about 2%

"* Due to this reduction the filament temperature rises about 20 K resulting in an... [increase] of light efficiency of about 8,8%" (2, p. 149)

Key Words: Technological Experiments, Halogen Lamps, Halogen Cycle, Heat Transfer, Buoyancy-Driven Convection, Gaseous Convection, Thermal Distribution, Thermal Environment More Extreme Than Predicted, Radiation, Electric Field, Vapor Deposition, Gas Pressure, Heat Pipes, Vacuum

Number of Samples: one
Sample Materials: Ellipsoidal mirror and halogen lamp. Instead of a sample, a photocell was inserted within the focus.
Container Materials: not applicable

Experiment/Material Applications:
This investigation was designed to test furnace components that would later be used in space processing hardware.

References/Applicable Publications:
(1) Cargo Systems Manual: GAS Annex for STS 41-G, JSC-17645 41-G, September 4, 1984. (very short description; preflight)

(2) Schmitt, G. and Stapelmann, J.: Halogen Lamp Experiment, HALEX. In NASA Goddard Space Flight Center's 1985 Get Away Special Experimenter's Symposium, October 8-9, 1985, pp. 141-149, NASA CP-2401. (post-flight)

(3) Space Shuttle Mission 41-G NASA Press Kit, October 1984, p. 23. (short description; preflight)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report # EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special Canister mission history)

(5) Input Received from K. Kemmerle, December 1989.

(6) Input Received from G. Schmitt (Kayser-Threde), July 1993.

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Experiment Origin: USA
Mission: STS Launch #3, STS-003 (STS OFT-3, Columbia)
Launch Date/Expt. Date: March 1982
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Pallet Instrument: OSS-1 Pallet, STS Payload Bay
Processing Facility: Thermal Canister
Builder of Processing Facility: Grumman Aerospace Corporation, Bethpage, Long Island, New York

Experiment:
Thermal Canister Experiment (TCE)

The orbiter bay of the Space Shuttle, which can be used to house several types of experiments and instruments, is subjected to extreme thermal environment conditions, ranging from +100 ^{o}C (full sun exposure) to -100 ^{o}C (shadow). In an effort to protect experiments from this extreme environment, coatings, insulation, and/or heaters have been used for thermal control in the bay. Each time the shuttle flies in a different orbital attitude, experiments are often redesigned to handle the different thermal environment. If a thermal enclosure was created which provided protection for instruments from the widely varying environments, then simpler designs (with limited maintenance between flights) would be realized.

This space shuttle STS-003 experiment was the first in a series of investigations designed by Ollendorf and/or McIntosh et al. to study the performance of heat pipes under low-gravity conditions. The specific objective of the experiment was to demonstrate temperature stability at various points within a canister while dissipating up to (1) 325 watts in cold orbiter attitudes (bay away from Sun) and (2) 100 watts in hot conditions (bay towards the Sun).

The Thermal Canister Experiment (TCE) consisted (in part) of a rectangular (3 meters high by 1 meter by 1 meter) enclosure constructed of aluminum. Thermal control was provided by a system of longitudinal, fixed conductance heat pipes. The heat pipes collected thermal energy from (1) internal electric heaters designed to simulate operating instruments and (2) direct and reflected sunlight. This heat was then conducted to variable conductance heat pipes which were connected to radiators mounted on the upper end of the canister. The radiators radiated the heat to space.

"The [fixed conductance] heat pipes are long, narrow, closed chambers with internal capillary wicking which provides pumping action. The wick is saturated with a volatile liquid (ammonia) in equilibrium with its vapor. Heat transport is established by applying heat at one end (the evaporator) and providing cooling at the other end (the condenser) with the heat being transferred at latent heat of vaporization. The liquid is then returned to the evaporator by capillary forces in the wick.

"The variable conductance heat pipes are more complex than the fixed conductance type in that they contain a noncondensible gas (nitrogen) stored in a reservoir at the condenser end of each pipe. As the temperature of the evaporator end of the pipe falls, a heating element raises the temperature of the reservoir, causing the gas to expand into the condenser, thus blocking the condenser region and effectively stopping heat pipe action. The length of the condenser rendered inactive depends on the temperature level along the pipe. Conversely, with increasing evaporator-end temperature, the gas will recede into the reservoir making more active area of the radiators available for heat rejection to space. The signal for activating the reservoir heaters is supplied through a feedback loop consisting of a temperature control sensor and either a hardware proportional controller or a computer-driven controller. The sensors are attached to the canister side walls or on simulated instruments located in two different zones separated by an insulating barrier. The simulators are either radiatively or conductively coupled to the canister walls." (1, p. 405) <Note: The exact meaning of some of the information in this paragraph was unclear to the editors.>

Primary and secondary objectives of the experiment were also detailed. The primary objectives were (1) to maintain a temperature of 15 +/- 2 ^{o}C on all panels of the canister and (2) to maintain temperature control under (a) all orbiter bay environmental conditions and (b) a range of internal power dissipations. The secondary objectives were to (1) demonstrate thermal control from 5 to 25 ^{o}C, (2) operate the system in the passive mode with a variation of +/- 5 ^{o}C about some nominal temperature (which depended on the thermal environment), and (3) demonstrate the performance of microprocessor-driven algorithms for thermal control.

Approximately 5 hours after the launch of STS-003, microprocessor control was initiated to maintain the canister wall temperature at 15 ^{o}C (during initial stabilization of the shuttle). "Unfortunately the microprocessor experienced reset problems which precluded the commanding of selected power, temperature, and control functions. It was decided to bypass the computer and use a backup hard wire system which utilized a limited number of relay commands. This enabled the experiment to proceed and the

majority of primary and secondary objectives to be met. A total of 11 steady-state data points were[sic] achieved during the seven day mission.... Control was either on the canister walls...or on the instrument simulators...." (1, p. 406)

Post-flight, it was reported that, during the space experiment, the set point variations ranged from 5 to 23 ^{o}C with power dissipation of 325 W (cold attitude with shuttle tail toward Sun) to 165 W (hot attitude with payload bay toward Sun). The canister maintained temperatures to within +/- 2 ^{o}C on all panels at any control point with spatial gradients of 4 ^{o}C. (During ground testing, gradients as high as 8 ^{o}C were observed and were attributed to the uneven distribution of fluids in the heat pipes caused by gravity.)

After switching control to the radiatively coupled instrument simulator and cycling the power duty, a variation similar to the one above was maintained. When the simulator was uncontrolled, a variation of +/- 5 ^{o}C with time was noted. Passive thermal control (control system deactivated) was also demonstrated where the TCE fell only to -5 ^{o}C.

It was also reported that the most important data was achieved during the transition periods when the shuttle was passing from one attitude to another. During four transitions, the canister walls were held at a constant temperature (constant power input) while the thermal environment changed drastically. Because of the limited heat rejection capability during the hot attitude, the TCE could not maintain the desired set-point temperature of 14 ^{o}C. However, after changing the set point to 23 ^{o}C, thermal stability was achieved.

"Although the microprocessor continued to reset throughout the mission due to unknown reasons, there were several intervals where the control algorithms could be invoked. This occurred at least four times and demonstrated that the canister and simulated experiments could be controlled utilizing the software built into the computer." (1, p. 406)

Data obtained from sensors mounted on the upper and lower segments of the radiator was used to determine the thermal flux impinging on the canister. It was reported that the average orbital flux absorbed by the canister was higher than expected. This was attributed to (1) higher thermal input from the shuttle cargo bay (cold and moderate attitudes) and (2) uncertainties about the canister coatings (hot attitude). Because of this higher flux in the moderate and hot attitudes, the control set-point temperature had to be adjusted for thermal stability.

<Note: Performance of the heat pipes system during shuttle re-entry and landing is also reported in Reference (1).>

It was concluded that the TCE achieved all its primary and most of its secondary objectives during the mission. The performance of the heat pipes exceeded ground testing and no heat pipe dryout was detected. "It is felt that through this flight test, the thermal canister concept has been proven and it is ready for operational use to house scientific instruments which will be flown on future Shuttle missions." (1, p. 409)

Key Words: Technological Experiments, Thermal Control, Heat Transfer, Heat Pipes, Wicking, Capillary Flow, Capillary Forces, Surface Tension, Fluid Management, Vaporization, Vapor Transport, Evaporators, Evaporation, Condensers, Condensation, Heat Radiators, Liquid/Vapor Interface, Phase Transition, Coated Surfaces, Hardware Malfunction

Number of Samples: one thermal system containing an unspecified quantity of fixed conductance heat pipes
Sample Materials: ammonia, nitrogen
(N*H*, N*)
Container Materials: aluminum heat pipes
(Al*)

Experiment/Material Applications:
See **Experiment** section (above) and McIntosh, STS-013 (this chapter).

References/Applicable Publications:

(1) Ollendorf, S.: Thermal Canister Experiment in OSS-1. Journal of Spacecraft and Rockets, Vol. 21, July-August 1984, pp. 405-409. (post-flight)

(2) Ollendorf, S. and Butler, D.: Results of Thermal Experiment Measurements on the Thermal Cannister Experiment and Get Away Special Enclosure. In Systematics General Corp., The Shuttle Environment Workshop, NASA CR-170496, February 1983, pp. A-275 - A-287. (post-flight; appears to be viewgraphs only)

(3) Input received from Experiment Investigator, June 1989 and August 1993.

(4) Ollendorf, S.: Recent and Planned Developments at the Goddard Space Flight Center In Thermal Control Technology. In Proceedings of the International Symposium on Thermal Systems for Space Vehicles, Toulouse, France, October 4-7, 1983, pp. 45-51. (post-flight)

(5) McIntosh, R. and Ollendorf, S.: A Thermal Canister Experiment for the Space Shuttle. In 3rd International Heat Pipe Conference, Palo Alto, California, May 22-24, 1978, Technical Papers, AIAA Paper #78-456. (preflight)

(6) Ollendorf, S.: Thermal Canister Experiment on OSS-1. AIAA 21st Aerospace Sciences Meeting, January 10-13, 1983, Reno, Nevada, AIAA Paper #83-0254. (post-flight)

(7) Harwell, W. and Ollendorf, S.: The Heat Pipe Thermal Canister. AIAA 15th Thermophysics Conference, Snowmass, Colorado, July 14-16 1980, AIAA Paper #80-1461. (preflight)

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Co-Investigator(s): None
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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
<Note: An Experiment Investigator indicated that this payload also flew on "STS #11." Although the investigator cited two references which may have detailed the results of the STS #11 flight, these papers could not be obtained prior to the publication of this experiment summary (see Note in "References/Applicable Publications" below).>
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Payload Bay, STS Deployed Satellite, DFVLR SPAS-01 Platform (SPAS was a small experiment carrier initially configured in the STS payload bay but later deployed into orbit by the Canadian Remote Manipulator Arm. The carrier was retrieved prior to the end of the mission.)
Processing Facility: Constant-conductance heat pipes, variable-conductance heat pipes (VCHP), and heat-pipe diodes
Builder of Processing Facility: Various, see **Experiment** section below

Experiment:
<u>Heat Pipe Experiment on SPAS</u>

Generally, all heat pipes operate on the same principle: the working fluid evaporates at the heat source (evaporator section) and the latent heat is transferred in the vapor phase where it recondenses at a heat sink (condenser). The condensate is returned to the evaporator (via capillary forces) through a porous wick material. The heat transport capacity of a heat pipe is determined by the effectiveness of the wick material. This effectiveness is greatly influenced by gravity. On Earth, the liquid excess needed to saturate the wick collects in the bottom of the pipe. In space, however, the excess liquid will collect elsewhere and may affect the performance of the heat pipe.

The objective of this STS-007 experiment was to study the low-gravity performance of three different heat pipe systems configured on the Shuttle Pallet Satellite SPAS-01 (see Reference (3) for a description of SPAS-01): (1) constant-conductance heat pipes, (2) variable-conductance heat pipes, and (3) heat pipe diodes.

A document published prior to the launch of STS-007 (Reference (2)) reported that nine heat pipe experiments were to be performed during the SPAS-01 mission. Two of the experiments involved constant-conductance artery heat pipes (IA1 and IA2), four involved gas-controlled variable-conductance heat pipe (VCHP) radiators (DS R1, SA1, DS R2, and DS R3), two involved liquid trap diodes (ID1 and DS D1), and one involved a gas diode (DS D2). This preflight document described the expected experimental setup and goals of each of the nine investigations. A summary of each (as provided by Reference (2)) is presented in the following paragraphs.

IA1 and IA2: Each apparatus was provided by IKE/ESTEC and had "...stainless-steel artery designs...capable of transporting about 150 W when horizontal. Heat is supplied to the evaporators according to a pre-programmed power profile, allowing the load to be increased progressively from zero to 150 W. Should the heat transport limit of the pipe be lower than 150 W, the resulting dry-out and rapid rise in evaporator temperature is detected by the experiment control and data system and the unit is switched off. The heat transported by the pipes is absorbed by phase-change-material thermal capacitors (PCM cells) containing eicosane wax and attached to the condenser sections. A cooling time of several hours is provided for the wax to refreeze between experiment cycles. The experiments are each instrumented with eight thermistors to provide temperature data for control and for transmission to the ground." (2, p. 78) The only difference between the IA1 and IA2 experiments was to be the shorter evaporator length of the IA2 apparatus.

DS R1 and SA1: Each of these experiments was to contain a gas-controlled, variable-conductance heat pipe with an electrically heated evaporator section. Cooling of the condenser section was to be accomplished using a space-viewing radiator. The DS R1 heat pipe (designed by Dornier Systems) was comprised of extruded aluminum with an axial-groove wick structure. The pipe was to be equipped with eleven thermistors for control and monitoring. The wick structure was to be attached to a slotted radiator. During the mission, the heat load for the DS R1 experiment was to be stepped up to 20 W and then stepped back down. Each step was to be held for several hours allowing an assessment of the (1) external environment effects and (2) radiator and VCHP performance.

The SA 1 heat pipe (supplied by Societe Anonyme Belge de Constructions Aeronautiques (Sabca), Brussels) was constructed of stainless steel with a simple artery wick structure. The gas reservoir was to be cooled by direct radiation to space. During the mission, the heat load for the SA 1 experiment was to be stepped more often and with smaller steps than the DS R1 experiment. This would allow exploration of the heat transport

capacity and VCHP control function. The SA 1 heat pipe was to be equipped with twelve control and monitoring thermistors.

DS R2 and DS R3: The main task of these VCHP radiators was to remove heat from the experiment-support plate (ESP) which was to act as a heat sink for the DS D1 and DS D2 diodes, the control and data system, and the IA1 and IA2 PCM cells. The DS R2 apparatus (provided by Dornier Systems) consisted of one gas-controlled VCHP and the DS R3 (also provided by Dornier Systems) consisted of two gas-controlled VCHP's. All three VCHP's were similar in design to that of DS R1. The evaporator sections were to be directly attached to the ESP (heat source). The heat pipes were to control the ESP temperature to within a few degrees of 30 ^{o}C.

DS D1, DS D2, and ID1: All three of these experiments used heat-pipe diodes. Heat-pipe diodes are one-way heat pipes which use blockage techniques during the reverse or shut-down mode. These blockage techniques are either (1) non-condensible gases or excess liquid used to block the vapor space or (2) liquid traps into which the working fluid condenses. DS D1 (provided by Dornier Systems) was a liquid-trap diode experiment. Two identical diodes were to be mounted to a common plate at their evaporator (liquid trap) ends. The plate was to be equipped with a heater (forward mode heater). The free ends of the diodes were to be mounted to a PCM cell and the reverse-mode heater block, respectively. This would allow one diode to demonstrate forward mode operation with the heat transported to and absorbed by the PCM cell. The other diode demonstrated reverse mode operation. Ten thermistors were to monitor and control temperatures. ID1 (provided by IKE/ESTEC) was also a liquid-trap diode experiment which was designed to investigate the transient response of the diode to different condenser heat loads. This heat pipe was comprised of stainless steel and had a design based on that of the IA1 and IA2 heat pipes. Two heaters, one at the trap and the other located at the evaporator, were to be sequenced to establish a forward mode of operation. After switching off the two heaters, a heater at the condenser would be activated to initiate the shutdown. The evaporator and trap were to be cooled with a small radiator (with radiation to space). The ID1 experiment was to be equipped with ten thermistors. The DS D2 experiment (provided by Dornier Systems) used a gas diode heat pipe which basically consisted of a VCHP with a heater located at each end. In forward-mode operation, the gas is located at the condenser end in a reservoir. During reverse-mode operation, the gas is swept to the evaporator end and blocks the vapor space. This experiment was to use eight thermistors for performance monitoring.

<Note: Reference (2) contains a description of the expected control and data systems.>

No documents published after the STS-007 flight could be located which described (1) the actual flight experimental setup and procedure, or (2) the post-flight results. Although an Experiment Investigator indicated that two papers on this experiment were published, these documents could not be attained prior to the publication of this summary (see note in the References section below).

The Experiment Investigator briefly reported that: "Results as predicted with the exception of the Sabca heat pipes." (4)

<Note: It appears from the Investigator response that the heat pipes employed during this experiment were later flown on the ERS-1, ERS-2, and L-SAT (Olympus) spacecraft.>

Key Words: Technological Experiments, Heat Pipes, Thermal Control, Heat Transfer, Wicking, Porous Material, Capillary Flow, Capillary Forces, Surface Tension, Fluid Management, Vaporization, Vapor Transport, Evaporators, Evaporation, Condensers, Condensation, Heat Radiators, Phase Transition, Free-Flying Satellite

Number of Samples: Unknown; preflight documents detailed nine heat pipes.
Sample Materials: working fluids: NH_3, Freon 21, Freon 11.
(N*H*)
Container Materials: IA1, IA2, SA1, and ID1: stainless steel; DS R1: aluminum; DS R2 and DS R3: unknown, possibly aluminum; DS D1: unknown; DS D2: unknown
(Al*)

Experiment/Material Applications:
Satellites such as the Marecs maritime communications satellite currently use heat pipes for thermal control. Heat pipes are envisioned for used on several vehicles including the Franco-German TV-Sat and on the European Space Agency's L-SAT (Olympus) and ERS-1 spacecraft.

References/Applicable Publications:

(1) Koch, H., Kreeb, H., and Savage, C.: The Heat Pipe Experiment on SPAS 01. Luft-und Raumfahrt, Vol. 5, 4th Quarter, 1984, pp. 133, 134, 136-141. (in German)

(2) Savage, C. J.: A European Heat-Pipe Experiment on the Second Flight of Space-Shuttle 'Challenger.' In ESA Bulletin, No. 35, August 1983, pp. 76-81. (preflight)

(3) Davidts, D.: The Shuttle Pallet Satellite System. Journal of the Astronautical Sciences, Vol. 28, No. 3, July-September 1980, pp. 283-298. (preflight, SPAS description)

(4) Input received from Experiment Investigator, July 1993.

<Note: The Experiment Investigator indicated that two additional papers were given at the International Heat Pipe Conference, Tsu Kuba, Japan, 1985. However, the editors of this document could not obtain these papers prior to the publication of this summary.>

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Experiment Origin: USA
Mission: STS Launch #8, STS-008 (STS 31-D, Challenger)
Launch Date/Expt Date: August 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Payload Bay
Processing Facility: Proof of concept heat pipe radiator
Builder of Processing Facility: Grumman Space Systems, Bethpage, New York

Experiment:
Heat Pipe Radiator

This STS-008 experiment was the first in a series of investigations designed by Rankin et al. to study the performance of a high-capacity, monogroove heat pipe radiator prototype under low-gravity conditions.

For several years prior to the experiment, NASA-Johnson Space Center (NASA-JSC) had investigated heat pipe technology for use in space radiator systems. The ultimate goal was to develop a simple but highly reliable (and survivable) space radiator system suitable for long-duration missions. One of the concepts which was developed was called the Space Constructible Radiator (SCR).

Under the SCR program, the high-capacity monogroove or dual-passage heat pipe was developed. Although full size low-gravity testing was planned, the cancellation of the TDRS-B payload on the STS-008 mission provided an earlier opportunity for a scaled-down version of the system. Therefore, in less than four months, a flight experiment was conceived, designed, fabricated, tested, and integrated into shuttle cargo bay. The specific objective of this flight experiment was to demonstrate the low-gravity performance of a monogroove heat pipe.

The operating fluid for the STS-008 heat pipe system was Freon-21, although the monogroove heat pipe system was developed for ammonia. "...Freon-21 was used in the STS-8 hardware due to its five times lower vapor pressure and the fact that an early, lower-strength version of the heat pipe extrusion was being used." (1, p. 2) (See References (1) and/or (5) for details of pre-flight pipe burst test results.)

<Note: Not all of the Applicable Publications listed below could be located prior to the publication of this experiment summary. The following brief description of the employed heat pipe system was obtained from References (1) and (5).> The heat pipe radiator experiment apparatus consisted of a single U-shaped monogroove heat pipe which was bonded to a radiating fin. Heat input was provided by two thermostatically protected electric heaters (30 watts and 70 watts) which were attached to the underside evaporator flanges. Heat rejection was achieved through a double-sided aluminum radiator (3.1 mm thick) bonded to the condenser flange with conductive epoxy.

<Note: Although Reference (5) provided a figure of the STS-008 heat pipe configuration (see Figure 4), a more detailed (written) description of the heat pipe was not provided in References (1) and (5). From the provided figure and a description of the monogroove heat pipe design found in a reference related to Rankin's later STS-29 heat pipe experiment, it appears that the STS-008 heat pipe used no moving parts, but rather works through the convection currents of the working liquid:

Electric heaters warm one end of the radiator, turning the working liquid into vapor which transports the heat through the length of the pipe, where fin radiates it into space. The fin is cooled by the space environment, and the working fluid is, in turn, condensed and recirculated.

"Two small pipes run through the center of the radiator down its length, branching out like the tines of a fork at the end that receives heat, called the evaporator. The top pipe holds the vaporized ammonia; the bottom holds liquid ammonia. In the evaporator portion, a fine wire mesh wick, which works along the sample principles as the wick of an oil lamp, pulls the liquid ammonia from one pipe to the other, where it vaporizes. Small grooves allow the condensed ammonia to drop back to the bottom pipe." (7, p. 13)>

STS-008 documentation reported that "Since time precluded using shuttle systems for data acquisition, calibrated temperature-sensitive liquid crystal films which change color were used to monitor temperatures in the evaporator and condenser sections. Five different types of films were used, providing a temperature sensitivity range of 20 to 45 $^{\circ}$C. Each film was capable of a 5 $^{\circ}$C range with color variation of blue to red.... [R]eal-time visual observations were made through the aft flight deck windows and 35 mm photographs were taken by the astronauts." (1, p. 2) (See References (1) and/or (5) for descriptions of liquid crystal films.) As a backup, seven temperature indicating decals (Tempilabel), which permanently change color in response to a particular temperature, were used to establish a record of the

evaporator temperature.

During the STS-008 experiment, the space shuttle operated with essentially an Earth-oriented cargo bay ("+ ZLV orbit"). Electrical power (70 W) was applied to the system approximately 24 minutes after orbital dawn. Color changes in the liquid crystal films were observed within 25 minutes (see References (1) and/or (5)). The color pattern indicated that a temperature difference of approximately 5 ^{o}C existed between the evaporator and condenser sections with uniform temperatures along each section. (The 5 ^{o}C temperature difference had been predicted by thermal modeling.)

It was reported that the experiment operated in a stable mode for 2 hours and 35 minutes at a single 70 W power setting. After this time, the system was turned off. Post-flight examination of the Tempilabel decals revealed a maximum temperature of 49 ^{o}C for the evaporator. This result confirmed proper operation of the heat pipe for the entire experiment.

It was concluded that this experiment demonstrated the successful operation of the monogroove heat pipe radiator under low-gravity conditions. It was also reported that no priming or operating problems were observed at any time during the experiment.

Key Words: Technological Experiments, Heat Pipes, Thermal Control, Heat Transfer, Heat Radiators, Vaporization, Gas Pressure, Vapor Transport, Evaporators, Evaporation, Condensers, Condensation, Phase Transition, Fluid Management, Wicking, Capillary Flow, Capillary Forces, Convection, Liquid/Vapor Interface, Liquid Crystals

Number of Samples: one heat pipe
Sample Materials: working fluid: Freon-21
Container Materials: heat pipe: unknown; radiator: aluminum

Experiment/Material Applications:
See Experiment summary (above).

References/Applicable Publications:

(1) Alario, J. P.: Monogrove [sic] Heat Pipe Radiator Shuttle Flight Experiment: Design, Analysis and Testing. Fourteenth Intersociety Conference on Environmental Systems, San Diego, California, July 16-19, 1984. (post-flight report)

(2) Alario, J., Haslett, R. and Kosson, R.: The Monogroove High Performance Heat Pipe. AIAA Paper #81-1156.

(3) Alario, J., Brown, R., and Kosson, R.: Monogroove Heat Pipe Development for Space Constructible Radiator System. AIAA Paper #83-1431.

(4) Alario, J. et al.: Space Constructible Radiator Prototype Test Program. AIAA Paper #84-1793. (ground testing)

(5) Rankin, J. G.: Integration and Flight Demonstration of a High Capacity Monogroove Heat Pipe. AIAA Paper #84-1716. (post-flight report)

(6) Input received from Project Engineer, J. P. Alario, August 1993.

(7) NASA Space Shuttle Mission STS-29 Press Kit, March 1989, pp. 13-15. (pre-STS 29)

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Experiment Origin: USA
Mission: STS Launch #28, STS-29 (Discovery)
Launch Date/Expt Date: March 1989
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Payload Bay/Starboard Sill
Processing Facility: Space Station Heat Pipe Advanced Radiator Element (SHARE)
Builder of Processing Facility: Unknown

Experiment:
Space Station Heat Pipe Advanced Radiator Element (SHARE)

<Note: Information for this Experiment summary was obtained from a NASA press release which was published prior to the launch of STS-29. No other publications which discussed the objectives, experimental setup, or results could be located at this time.>

This STS-029 experiment was the second in a series of investigations designed by Rankin et al. to study the performance of a high-capacity, monogroove heat pipe radiator concept under low-gravity conditions (see Rankin STS-008). The specific objective of the experiment was to test a potential, heat-pipe cooling system for the Space Station Freedom.

"The heat pipe method uses no moving parts and works through the convection currents of ammonia. Three electric heaters will warm one end of the 51-foot long SHARE [Space Station Heat Pipe Advanced Radiator Element]. The heaters turn liquid ammonia into vapor which transports the heat through the length of the pipe, where a foot-wide aluminum fin radiates it into space. The fin is cooled by the space environment, and the ammonia is, in turn, condensed and recirculated.

"Two small pipes run through the center of the radiator down its length, branching out like the tines of a fork at the end that receives heat, called the evaporator. The top pipe holds the vaporized ammonia; the bottom holds liquid ammonia. In the evaporator portion, a fine wire mesh wick, which works along the sample principles as the wick of an oil lamp, pulls the liquid ammonia from one pipe to the other, where it vaporizes. Small grooves allow the condensed ammonia to drop back to the bottom pipe.

"The radiator for SHARE weighs about 135 pounds, but with its support pedestals, support beam, heaters and instrumentation package, the total experiment weighs about 650 pounds.

"Crew members will switch the heaters on by using controls located on the aft flight deck. Each of the experiments two 500-watt heaters and single 1000-watt heater is controlled individually and will be switched on in turn, applying heat that will increase steadily in 500-watt increments up to a maximum of 2000 watts.

"The experiment will be activated for two complete orbits in two different attitudes, the first with the payload bay toward Earth and the second with the orbiter's tail toward the Sun. The heaters go through a complete 500-watt to 2000-watt cycle for each activation. This will simulate the heat that needs to be dissipated from the Space Station, and the two attitudes will provide data on the heat pipe's operation in different thermal environments.

"Other information may be obtained during STS-29 if time permits, including a test of the heat pipe's minimum operating temperature, thought to be about minus 20 degrees Fahrenheit, and a test of its ability to recover from acceleration." (2, p. 13)

Key Words: Technological Experiments, Heat Pipes, Thermal Control, Heat Transfer, Heat Radiators, Vaporization, Vapor Transport, Evaporators, Evaporation, Condensers, Condensation, Phase Transition, Fluid Management, Wicking, Capillary Flow, Capillary Forces, Convection, Liquid/Vapor Interface, Surface Tension, Acceleration Effects

Number of Samples: one experimental setup
Sample Materials: working fluid: ammonia
(N*H*)
Container Materials: heat pipe: unknown; radiator: aluminum

Experiment/Material Applications:
See Rankin, STS-008.

References/Applicable Publications:

(1) Hartsfield, J.: SHARE: Ammonia-Powered "Air Conditioner" Gets Flight Test on STS-29. NASA Activities, March 1989, Vol. 20, No. 3, p. 13. (preflight)

(2) NASA Space Shuttle Mission STS-29 Press Kit, March 1989, pp. 13-15. (preflight)

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Principal Investigator(s): Walden, V. (1)
Co-Investigator(s): Megill, L. R. (Payload Manager) (2), Utah Section of American Institute of Aeronautics and Astronautics (Purchaser and Donor) (3)
Affiliation(s): (1) During STS-011: Utah State University, Logan, Utah, Currently: University of Washington, Seattle, Washington; (2) During STS-011: Utah State University Faculty, Logan, Utah, Currently: ARME Enterprises, Hyrum, Utah; (3) Utah

Experiment Origin: USA
Mission: STS Launch #10, STS-011 (STS 41-B, Challenger)
Launch Date/Expt. Date: February 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: College Student Experiment
NASA Get Away Special (GAS) canister G-008
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of this experiment within G-008: Utah State University, Logan, Utah
Processing Facility: Heat Pipe
Builder of Processing Facility: Utah State University, Logan, Utah

Experiment:
<u>Heat Pipe Fluid Flow Experiment, #1</u>

This experiment was the first in a series of investigations designed by Walden et al. to study heat pipe fluid flow. The experiment was one of four investigations housed within the G-008 Get Away Special canister during STS-011. Two other experiments (of the four) were applicable to this data base (see Alford, STS-011 (Chapter 18); Gerpheide, STS-011 (Chapter 16)).

Reportedly, the long-term objective of the experiment was to determine if the fluid dynamics of a heat pipe fluid/wicking system could be (later) used to perform electrophoretic separations. The experiment was not configured to achieve electrophoresis but rather was designed to illustrate the properties of a heat pipe working fluid as it flowed through the wicking material.

The experimental apparatus included a 12-inch long heat pipe constructed of 1-inch diameter glass tubing. The working fluid of the pipe was water and the wicking material was chromatography paper. The water was dyed such that heat pipe operation could be verified visually.

<Note: No further information concerning the experimental setup or the expected inflight operational scenario could be located at this time.>

The Principal Investigator reported that the heat pipe fluid flow experiment failed during this mission because the apparatus was improperly reset during ground testing. As a result of this improper setting, only 6 minutes of data were gathered. It was believed that during the time this data were gathered, the experiment was not in orbit. Therefore, it was reported that no useful data were obtained.

Reference 6 further reported that "A battery pack latching relay stuck closed prelaunch, resulting in battery drain before launch." (6, p. 29)

Key Words: Technological Experiments, Heat Pipes, Wicking, Capillary Flow, Capillary Forces, Surface Tension, Liquid Transfer, Separation of Components, Electrophoresis, Battery Drain

Number of Samples: one
Sample Materials: Working fluid: dyed water; wicking material: chromatography paper
Container Materials: The heat pipe was made from glass tubing.

Experiment/Material Applications:
Reportedly, this preliminary experiment was designed to investigate the possibility of using heat pipe technology to perform electrophoresis. Low-gravity electrophoretic processing is expected to yield separated samples of higher quality than similar separations obtained on Earth.

References/Applicable Publications:
(1) Cargo Systems Manual: GAS Annex for STS-11, JSC-17645 Annex STS-11, December 2, 1983. (mentions G-008 but does not detail this experiment; preflight)

(2) Input received from Principal Investigator, V. Walden, August 1989.

(3) Getaway Special Payloads (STS-11). In Goddard Space Flight Center's Engineering Newsletter, Vol. 2, No. 3, April 1984, Published by the Engineering Directorate, pp. 8-9. (very short description)

(4) STS-11 Getaway Special Payload Descriptions, NASA News, NASA GSFC, 1984. (post-flight)

(5) STS 41-B Tenth Space Shuttle Mission, Press Kit, February 1984, p. 28. (brief mention of experiment; preflight)

(6) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(7) STS-11 GAS Payloads. NASA Goddard Space Flight Center Engineering Newsletter, April 1984.

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Principal Investigator(s): Walden, V. (1)
Co-Investigator(s): Megill, L. R. (Payload Manager) (2), Utah State University/Jensen, B. C. (Contributor/Customer) (3)
Affiliation(s): (1) Graduated June 1984 from Utah State University, Logan, Utah, During STS-017: Quantic Industries, San Carlos, California, Currently: University of Washington, Seattle, Washington; (2) During STS-017: Utah State University Faculty, Logan, Utah, Currently: ARME Enterprises, Hyrum Utah; (3) Logan, Utah

Experiment Origin: USA
Mission: STS Launch #13, STS-017 (STS 41-G, Challenger)
Launch Date/Expt. Date: October 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: College Student Experiment
NASA Get Away Special (GAS) canister G-518
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-518: Utah State University, Logan, Utah
Processing Facility: Heat Pipe
Builder of Processing Facility: Unknown, possibly Utah State University, Logan, Utah

Experiment:
Heat Pipe Fluid Flow Experiment, #2

This experiment was the second in a series of investigations designed by Walden et al. to study heat pipe fluid flow (see Walden, STS-011). The experiment was one of four investigations housed within the G-518 Get Away Special canister during STS-017. Three other experiments (of the four) were applicable to this data base (see Kitaura, STS-017 (Chapter 2); Thomas, S., STS-017 (Chapter 12); "Solder Flux Separation," STS-017 (Principal Investigator unknown (this chapter))).

The specific objective of this experiment was to determine if the fluid dynamics of a heat pipe fluid/wicking system could be used to perform electrophoretic separations. The experiment was not configured to achieve electrophoresis but was designed to illustrate the properties of a heat pipe working-fluid as it flowed through the wicking material.

The experimental apparatus included a 12-inch long heat pipe constructed of 1-inch diameter glass tubing. The working fluid of the pipe was water; the wicking material was chromatography paper. The water was dyed such that heat pipe operation could be verified visually.

A document released prior to the launch of STS-017 (Reference (2)) further detailed the expected experimental setup and in-flight operation of the fluid flow system. Reportedly, the glass tube heat pipe was to be partially evacuated and configured with (1) a heater at one end and (2) a paraffin heat sink at the other end. During the experiment, (1) the heater was to vaporize a small amount of water within the tube, (2) the vapor was to recondense at the heat sink, and (3) the recondensed water was to return to the hot end of the pipe via the wicking material. Although it was noted that the system was expected to separate the dye from the water, further discussion of this separation was not provided.

The Principal Investigator reported that during the mission, the experiment operated through to completion but the data were not usable. It was determined that either the experiment batteries were too low to properly record the data, or a software error existed in the computer controller.

No further information concerning this experiment could be located at this time.

Key Words: Technological Experiments, Heat Pipes, Wicking, Capillary Flow, Capillary Forces, Surface Tension, Liquid Transfer, Phase Transition, Vaporization, Condensation, Liquid/Vapor Interface, Fluid Management, Separation of Components, Electrophoresis, Battery Drain, Processing Difficulties

Number of Samples: one
Sample Materials: Working fluid: dyed water; wicking material: chromatography paper
Container Materials: The heat pipe was made from glass tubing.

Experiment/Material Applications:
Please see Walden, STS-011.

References/Applicable Publications:
(1) Cargo Systems Manual: GAS Annex for STS 41-G. JSC-17645 41-G, September 4, 1984. (short description; preflight)

(2) NASA Space Shuttle Mission 41-G Press Kit, October 1984, pp. 24-25. (preflight)

(3) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(4) G-518 Payload Accommodations Requirements, NASA Goddard Space Flight Center, March 20, 1984.

(5) Press Release for G-518, Utah State University, Logan, Utah, 1984.

(6) Input received from Principal Investigator V. Walden, August 1989.

(7) Letter from V. Walden to L. Rex Megill dated December 3, 1984.

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Co-Investigator(s): None
Affiliation(s): (1,2) McDonnell Douglas Astronautics Company, St. Louis, Missouri

Experiment Origin: USA
Mission: Launched: STS Launch #11, STS-013 (STS 41-C, Challenger); Returned: STS-032 (Columbia)
Launch Date/Expt. Date: April 1984. The experiment, which was on the LDEF free-flying facility, orbited the Earth for 6 years and then was returned via the space shuttle in January 1990.
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Experiment within the Long Duration Exposure Facility (LDEF) (a STS Deployed Satellite)
Processing Facility: Cascaded Variable-Conductance Heat Pipe (CVCHP)
Builder of Processing Facility: McDonnell Douglas Astronautics Company, St. Louis, Missouri

Experiment:
Cascade Variable-Conductance Heat Pipe (LDEF A0076)

The Long Duration Exposure Facility (LDEF) was a free-flying cylindrical structure (30 ft. long and 14 ft. in diameter) placed in orbit by the U.S. space shuttle at an altitude of 257 nautical miles and an inclination of 28.5 degrees. The structure contained 57 science and technology experiments located in trays mounted on the exterior of the structure. LDEF was to be retrieved after approximately 9 months. However, the structure remained in orbit for nearly 6 years because U.S. shuttle flights were delayed following the loss of the space shuttle Challenger. LDEF was eventually retrieved at an altitude of approximately 180 nautical miles.

This LDEF experiment was designed to examine the low-gravity performance of a cascaded variable-conductance heat pipe (CVCHP). The specific objective of the experiment was to determine if such a heat pipe configuration could provide precise temperature control of future space vehicles (within +/- 0.3 ^{o}C). These space vehicles are expected to (1) operate for extended periods of time and (2) have widely varying power inputs.

The heat pipe configuration used two series-connected dry reservoir VCHPS. Ammonia was used as the working fluid and nitrogen as the control gas.

In a dry gas reservoir, the control gas (noncondensible) is isolated from the ammonia in the heat pipe. The reservoir is thermally connected to the evaporator to maintain (1) the gas above

the evaporator temperature and (2) the reservoir at a constant temperature.

Reportedly, "In initial [theoretical] analysis, a single VCHP could not maintain a precise temperature control in the widely ranging heat loads and environments [of space], but could easily maintain a control of +/-3.0 ^{o}C. [Thus] a second, series connected (i.e. cascaded) VCHP was added [to the LDEF experiment configuration] to provide precise temperature control. Using the +/-3.0 ^{o}C control of the 'coarse' control VCHP as the sink temperature, the 'fine' control VCHP could provide +/-0.3 ^{o}C control without requiring an excessive reservoir size." (3, p. 1)

In order for the dry reservoir concept to be successful, the ammonia vapor had to be kept out of the noncondensible gas reservoir. If ammonia entered the reservoir, a rise in the temperature set point would occur. A long capillary tube was located between the heat pipe and reservoir to prevent diffusion of the ammonia into the reservoir. Ground-based, short-duration thermal vacuum tests demonstrated that this concept was successful. However, a long-term space test was required to determine the drift in temperature set point caused by diffusion.

The CVCHP did not require electrical power to operate but did require a heat throughput (e.g., equipment waste heat). On LDEF, this was accomplished by using (1) a black chrome solar collector for heat input (simulating for example, an equipment heat load) and (2) a silver/teflon radiator for heat rejection. The experiment was located on the leading edge (row 9) of LDEF, which provided a widely varying thermal environment (full solar to full shade) every orbit. Six thermistors provided thermal data, which was recorded in the data system of an adjacent LDEF experiment. Data were collected about every 2 hours for approximately 45 days (until battery drain). (References (4) and (5) provide detailed descriptions of the heat pipe.)

During the initial period of LDEF deployment (approximately 6 days), LDEF was in a low angle orbit and, therefore, was subjected to widely varying environmental conditions. During this time, the thermal data indicated that both VCHPs maintained an almost constant temperature. The thermal data from the fine control VCHP indicated a slight temperature variation in response to the varying solar collector input. The thermal data from the coarse control VCHP indicated a nearly constant temperature with a minimum temperature recorded between days 10 and 20. This corresponded to the period of minimum thermal environmental conditions. The coarse control VCHP reservoir showed very little change throughout the mission.

Reportedly, "One of the key goals of the flight experiment was to determine if there would be a long term upward drift due to diffusion of ammonia into the reservoir, but the data indicated no major temperature changes. Both VCHPs reached their minimum temperatures between day 10 and 20 and then rose slightly through day 45, but it was impossible to determine if this very slight increase was due to a very small amount of diffusion or due to the warmer environment.... The very successful operation during the 45 days of data recording showed that the capillary tube did a very good job of preventing ammonia from entering the reservoir, but a significant amount of additional data would be required to extrapolate to see if there exists a small temperature setpoint rise over an extended mission." (3, p. 6)

It was concluded that the CVCHP demonstrated successful temperature control of +/- 0.3 $^{\circ}$C over the widely varying environment during the LDEF mission. "The flight data indicated that only one VCHP was required to maintain precise temperature control. Post-flight tests indicated upward shifts in both VCHP set points, but it was uncertain whether these shifts were caused by on-orbit diffusion or post-flight activities. Even if all the shift accrued in orbit, the average drift rate was less than 1 $^{\circ}$C per year." (3, p. 9)

<Note: Reference (4) also reported results from the erosion of the external surfaces caused by the orbital environment (e.g., atomic oxygen).>

Key Words: Technological Experiments, Heat Pipes, Thermal Control, Solar Heating, Evaporators, Capillary Flow, Capillary Forces, Heat Transfer, Heat Radiators, Diffusion, Free-Flying Satellite

Number of Samples: one cascade variable conductance heat pipe setup

Sample Materials: Working fluid: ammonia; control gas: nitrogen (N*H*, N*)

Container Materials: unknown

Experiment/Material Applications:
"Precision temperature control which requires no electrical power for operation can be beneficial in a number of spacecraft applications for controlling temperature sensitive components." (3, p. 1)

References/Applicable Publications:

(1) Grote, M. G. and Calhoun II, L. D.: Cascade Variable-Conductance Heat Pipe (A0076). In The Long Duration Exposure Facility (LDEF) Mission 1 Experiments, NASA SP-473, pp. 66-69, 1984. (preflight)

(2) Input received from Experiment Investigator, June 1988 and July 1993.

(3) Grote, M. G.: Results from the Cascaded Variable Conductance Heatpipe Experiment on LDEF. AIAA Paper #91-1356, to be published June 25, 1991, 31 pp. (post-flight, received from Grote)

(4) Calhoun, L. D. and Grote, M. G.: The Cascaded Variable Conductance Heatpipe Experiment on LDEF. AIAA Paper #AIAA-81-1157, 16th Thermophysics Conference, Palo Alto, California, June 23-25, 1981, 8 pp. (preflight, heat pipe description)

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Co-Investigator(s): Unknown
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Experiment Origin: USA
Mission: Launched: STS Launch #11, STS-013 (STS 41-C, Challenger); Returned: STS-032 (Columbia)
Launch Date/Expt. Date: April 1984. The experiment, which was on the LDEF free-flying facility, orbited the Earth for 6 years and then was returned via the space shuttle in January 1990.
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Experiment within the Long Duration Exposure Facility (LDEF) (a STS Deployed Satellite)
Processing Facility: Transverse Flat-Plate Heat Pipes
Builder of Processing Facility: Unknown

Experiment:
Transverse Flat-Plate Heat Pipe Experiment (S1005)

The Long Duration Exposure Facility (LDEF) was a free-flying cylindrical structure (30 ft. long and 14 ft. in diameter) placed in orbit by the space shuttle at an altitude of 257 nautical miles and an inclination of 28.5 degrees. The structure contained 57 science and technology experiments located in trays mounted on the exterior of the structure. LDEF was tc be retrieved after approximately 9 months. However, the satellite remained in orbit for nearly 6 years because shuttle flights were delayed following the loss of the space shuttle Challenger. LDEF was eventually retrieved at an altitude of approximately 180 nautical miles.

This LDEF experiment was designed to demonstrate the low-gravity performance of existing heat pipe thermal control technology. The specific objectives of the experiment were to (1) provide an "integral temperature control/mounting panel" for electronic equipment, scientific instruments, and experiments, (2) evaluate the thermal performance of heat pipe modules under low-gravity conditions, and (3) correlate retrieved flight data with data from pre- and post-flight thermal vacuum tests. Evaluation of the thermal performance (objective #2) included determining (1) the transport capability of the pipes, (2) temperature drops in the system, (3) the ability of the pipes to maintain temperature over varying duty cycles and environments, and (4) pipe performance degradation.

The experiment utilized current transverse flat-plate heat pipe technology. "A transverse heat pipe is a variable-conductance heat pipe (VCHP) which can handle relatively large thermal loads. It was developed to circumvent the gas bubble artery blockage problem associated with conventional artery wick designs which limited their capacity to small loads in the VCHP mode. In the basic design of a transverse heat pipe, liquid flows in a direction transverse or perpendicular to the vapor flow. Temperature control is achieved by using conventional noncondensible-gas techniques." (1, p. 74)

Three transverse flat-plate heat pipe modules were contained in one of the LDEF experimental trays. Heat was supplied to the evaporator side by 28-V lithium monofluorographite batteries. The batteries simulated various watt density equipment heat dissipaters. The heat was radiated to space from the outboard-facing radiator surface. <Note: Although the experiment objectives stated that the experiment was to provide integral temperature control for electronic equipment, scientific instruments and experiments, it appears that there were no such instruments or experiments associated with the heat pipe. The thermal loads, (as stated above) were supplied by batteries.> Thermal data were obtained via thermocouples and recorded on magnetic tape. The entire experiment was self-contained with respect to power supply, data storage, and on-orbit cycling. Power for data recording was provided by $LiSO_2$ batteries.

During the LDEF mission, the experiment had three "on-times", each lasting approximately 13 hours (8.6 orbits). Each "on-time" was divided into two 4.3 orbit heater input sub-periods lasting approximately 6.5 hours. The first "on-time" was initiated about 1 month after the shuttle launch, the second was 6.75 days later, and the third was 135 days after the shuttle launch.

After the LDEF had been retrieved and returned to Earth, the experiment package and all components were subjected to a complete systems checkout. It was determined that the batteries still contained power but were drained quickly, much like a discharging capacitor. The heater elements were cycled on/off to verify operation, the thermistor data was collected, and calibration was verified. It was reported that the experiment power and data (EPDS) components were within nominal operating parameters and that the experiment was returned in complete operational condition.

No other results from this experiment had been published when this experiment summary was prepared.

Key Words: Technological Experiments, Heat Pipes, Heat Transfer, Thermal Control, Evaporators, Heat Radiators, Vaporization, Fluid Management, Free-Flying Satellite

Number of Samples: three transverse heat pipes
Sample Materials: unknown
Container Materials: unknown

Experiment/Material Applications:
Heat pipes have been used on spacecraft for thermal control purposes. Little or no power consumption is expended using such heat pipes as the thermal control system.

References/Applicable Publications:
(1) Owen, J. W. and Edelstein, F.: Transverse Heat Pipe Experiment. In NASA Langley Research Center Long Duration Exposure Facility (LDEF), NASA SP-473, pp. 74-77. (preflight)

(2) Edelstein, F.: Transverse Flat Plate Heat Pipe Experiment. AIAA Paper #78-429, pp. 254-259. (preflight)

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Experiment Origin: USA
Mission: Launched: STS Launch #11, STS-013 (STS 41-C, Challenger); Returned: STS-032 (Columbia)
Launch Date/Expt. Date: April 1984. The experiment, which was on the LDEF free-flying facility, orbited the Earth for 6 years and then was returned via the space shuttle in January 1990.
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Experiment within the Long Duration Exposure Facility (LDEF) (a STS Deployed Satellite)
Processing Facility: Heat Pipe Experiment Package (HEPP)
Builder of Processing Facility: Unknown

Experiment:
<u>Low Temperature Heat Pipe Experiment Package (HEPP) (S1001)</u>

The pumping action of a heat pipe is driven by relatively weak gravity-independent, capillary forces. Reliable data concerning the performance of heat pipes is difficult to obtain on Earth because gravity-dependent forces acting on the system often overwhelm gravity-independent forces. Determining the role of capillary forces is further compounded on Earth when studying fluids which have relatively low surface tensions (e.g., low-temperature and cryogenic fluids).

This experiment was the second in a series of investigations designed by McIntosh and/or Ollendorf et al. to study the performance of heat pipes under low-gravity conditions (see Ollendorf, STS-003 (this chapter)). Specifically, the experiment was designed to investigate the performance of low-temperature (<190 K) heat pipes during the Long Duration Exposure Facility (LDEF) mission. The objectives of the experiment were to (1) determine the start up performance of low-temperature conventional and diode heat pipes under low-gravity conditions, (2) evaluate the low-gravity performance of the heat pipes over an extended period of time, (3) determine the low-gravity transport performance of the heat pipes, and (4) determine the forward conductance, turndown ratio, and transient behavior of the diode heat pipe.

The Long Duration Exposure Facility was a free-flying, cylindrical structure (30 ft. long and 14 ft. in diameter) placed in orbit by the space shuttle at an altitude of 257 nautical miles and an inclination of 28.5 degrees. The structure contained 57

science and technology experiments located in trays mounted on the exterior of the structure. LDEF was to be retrieved by the shuttle after approximately 9 months. However, the satellite remained in orbit for nearly 6 years because shuttle flights were delayed following the loss of the space shuttle Challenger. LDEF was eventually retrieved at an altitude of approximately 180 nautical miles.

The Heat Pipe Experiment Package (HEPP) was located in a tray which pointed toward deep space for the duration of the mission. Two heat pipes, a constant conductance heat pipe (CCHP) and a thermal-diode heat pipe, were coupled to a radiator cooler system. Both heat pipes used ethane as the working fluid. Also integrated with the radiator was a phase change material (PCM) canister which allowed thermal stability during transport tests (the PCM was N-heptane which has a melting point of 182 K). The canister provided a high-heat capacity (28 W-hr of latent heat) which allowed high-power heat pipe testing (e.g., 40 W for 40 minutes) at a constant temperature.

Multilayer insulation blankets and a shielding configuration were used to maximize radiation to deep space and protect the apparatus from impacts. Also located on the experiment tray were coated Kapton samples for investigation of atomic oxygen erosion. Standard LDEF power and data systems were used for data collection and recording. Power was provided by a dedicated solar-panel Ni-Cd battery system located in another tray.

Post-flight, it was reported "...that the HEPP cooled to a minimum of 190 K on a cyclic basis. The operation of the CCHP and the diode with a 1-watt heat load applied to each was demonstrated over the entire 13 month period of recorded data. However, the inability of the HEPP to cool below 180 K prevented the electronics from executing programmed test profiles. As a result transport tests and diode reverse mode tests were not conducted and of course the freezing and thawing of the PCM could not be achieved." (3, p. 99)

No other results from this experiment had been published at the time this experiment summary was written.

Key Words: Technological Experiments, Heat Pipes, Thermal Control, Capillary Flow, Capillary Forces, Surface Tension, Heat Transfer, Liquid Transfer, Heat Radiators, Solar Energy, Phase Transition, Cryogens, Free-Flying Satellite, Processing Difficulties

Number of Samples: two heat pipes
Sample Materials: Working fluid: ethane; phase change material: N-heptane
(C*H*)
Container Materials: unknown

Experiment/Material Applications:
"Although the majority of heat pipe applications to date have been related to ambient temperature operation, a number of applications for low temperature and cryogenic heat pipes have been identified. Cryogenic heat pipe radiant coolers could be used to augment or replace solid cryogen coolers in order to achieve longer life and/or reduce weight. The coupling of remote sensors to a centrally located cooler... has also been considered." (2, p. 418)

References/Applicable Publications:
(1) McIntosh, R., Jr., Ollendorf, S., and McCreight, C. R.: Low-Temperature Heat Pipe Experiment Package (HEPP) for LDEF (S1001). In The Long Duration Exposure Facility (LDEF) Mission 1 Experiments, NASA SP-473, pp. 70-71. (preflight).

(2) Suelau, H. J., Brennan, P. J., and McIntosh, R.: HEPP-A Low Temperature Heat Pipe Experiment Package Developed for Flight On-Board the Long Duration Facility (LDEF). In Third International Heat Pipe Conference, Palo Alto, California, May 22-24, 1978, Technical Papers, AIAA Paper #78-459, pp. 418-425. (preflight)

(3) McIntosh, R. and Brennan, P. J.: Long Duration Exposure Facility (LDEF) Low-Temperature Heat Pipe Experiment Package (HEPP). In First LDEF Post-Retrieval Symposium Abstracts, Kissimmee, Florida, June 2-8, 1991, NASA CP-10072, p. 99. (post-flight, abstract of conference presentation)

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Co-Investigator(s): Peterson, N. E., Jr. (2), National Aeronautics and Space Administration (NASA), Goddard Space Flight Center (3)
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Experiment Origin: USA
Mission: STS Launch #16, STS-023 (STS 51-D, Discovery)
Launch Date/Expt. Date: April 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) canister G-471
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-471: NASA Goddard Space Flight Center, Greenbelt, Maryland/OAO, Inc., Greenbelt, Maryland
Processing Facility: A miniaturized capillary pumped loop heat pipe system which included (1) two capillary pumped evaporators mounted in parallel, (2) integral heaters attached directly to the outer surfaces of the evaporators, (3) a fluid loop, and (4) a condenser plate.
Builder of Processing Facility: The OAO Corporation, Greenbelt, Maryland, built the miniaturized capillary pumped loops.

Experiment:
Capillary Pumped Loop (CPL)

Capillary Pumped Loop (CPL) heat pipe systems can possibly be used to provide thermal control of (1) the environment onboard an orbiting spacecraft or (2) scientific instruments or components onboard the craft. The CPL system employs a wick of porous material (high density polyethylene) which, via capillary forces, aids in the pumping of fluid in the closed loop heat pipe. The working fluid of the system is drawn through the wick to the metallic shell of the evaporator where heat is added and the liquid vaporizes. A pressure gradient produced by the evaporation process pumps the vapor to the condenser. At the condenser, the heat is removed from the vapor and the vapor condenses. The resultant liquid is returned via the wick to the evaporator where the cycle repeats.

This experiment was the first in a series of investigations designed by McIntosh et al. to demonstrate the capability of a capillary pumped loop system (operating in low gravity) to achieve thermal control.

Ground testing of an engineering model employing eight capillary pumps mounted in parallel indicated that the system had heat car-

rying capabilities of up to 6.4 kilowatts. Because the capillary pump operation is sensitive to the effects of gravity, space testing of a CPL system was proposed to evaluate the low-gravity performance of the heat pipe.

<Note: Reference (2), which provided the most detailed description of the CPL system, discussed both the STS-023 and STS-025 experimental setup and experimental sequence as if they were exactly the same on both missions (except for a change to the battery encasement for STS-025). Therefore, it was assumed that the expected experiment sequence as presented below (which was based on Reference (2)) applies to STS-023. However, as can be seen in editorial notes presented later in this experiment summary, other references (which were published prior to the launch of STS-023) do not correlate with the description provided in Reference (2).>

The experiment was developed using a significant amount of hardware which had flown in support of previous (essentially unrelated) GAS experiments. Thus, the size of the CPL system that could be examined was somewhat limited by the volume requirements of this available hardware. Nonetheless, a "mini-CPL" system was developed (measuring approximately 14" long by 14" wide by 4" high) which maintained most of the operating features of the ground-based system.

The mini-CPL system included (1) two capillary pumped evaporators mounted in parallel, (2) integral heaters attached directly to the outer surfaces of the evaporators, (3) a temperature-controlled, two-phase reservoir, (4) a fluid loop charged with ammonia, (5) a condenser plate (heat sink), and (6) various control electronics.

The importance of a two-phase reservoir was detailed. "By controlling the reservoir temperature, the loop temperature is controlled as well, since the saturation temperature of the working fluid is controlled at the reservoir. This means that the pumps (evaporators) stay at a relatively constant temperature regardless of temperature control in the loop. The loop temperature can be varied simply by raising or lowering the reservoir temperature to the desired level. Another salient feature of the reservoir is fluid inventory control. The reservoir can also be used for pressure priming of the pumps during startup operations." (2, p. 238)

The expected space experiment operating times were based on a (preflight) thermal control analysis of the CPL GAS payload. This analysis indicated that the payload could accommodate "...experiment heater cycles of up to 220 watts total (110 watts on each pump) for operating times of up to one hour, followed by a cool down period lasting approximately 10 hours." (2, p. 241)

Reportedly, the expected heating/cooling sequence of the payload was the following: "The experiment condenser was... [to initially] cool to approximately 5 ^{o}C, then the experiment heaters were [to be] activated. Since the power input... [was expected to exceed] the instantaneous heat rejection capability of the GAS top plate, the condenser temperature... [would increase]. When the condenser temperature... [approached] the CPL operating temperature of 29 ^{o}C it... [would no longer be able to] absorb any more heat and the system... [would] shut down and... cool down. The heater cycle... [would then be] repeated after the condenser... [cooled] back down to about 5 ^{o}C. During the flight, the cycles [were to be] repeated for the total mission time, approximately 120 hours." (2, p. 241)

<Note: A description of the expected experiment operating time as detailed in Reference (5) (published prior to the STS-023 Shuttle launch) differed somewhat to the operating time detailed directly above: "During the Shuttle flight, the experiment will be turned on within 24 hours of launch and continue for at least 60 hours and up to 96 hours, if possible." (5, p. 17)

Further, Reference (3) (also published prior to the STS-023 Shuttle launch) indicated that the working fluid was to be Freon-11, not ammonia.>

Post-flight analysis of the payload indicated that "...the GAS batteries that actuate the relay to activate the experiment failed so the CPL could not be turned on during flight. The failure was apparently due to a bad batch of batteries that failed under a combination of vacuum and cold temperatures, even though they had passed qualification testing." (2, p. 244)

Key Words: Technological Experiments, Heat Pipes, Thermal Control, Heat Transfer, Vaporization, Evaporators, Evaporation, Condensers, Condensation, Phase Transition, Wicking, Surface Tension, Capillary Flow, Capillary Forces, Liquid Transfer, Liquid/Vapor Interface, Fluid Management, Porous Material, Battery Failure

Number of Samples: one mini-CPL setup
Sample Materials: It appears that the working fluid was ammonia NH_3.(N*H*) However, one document (Reference (3), which was published prior to the flight) indicated that the working fluid was to be Freon-11.
(N*H*)

Container Materials: unknown

Experiment/Material Applications:
This research was initiated to demonstrate that CPL heat pipes could be used in a low-gravity environment to provide thermal control for scientific instruments and spacecraft. CPL thermal control systems may be placed on future spacecraft. "The capillary pumped-loop approach acquires and transports heat nearly isothermally for long transport distances and under a wide range of power levels." (7, p. 9)

Reportedly, ammonia was chosen as the working fluid because it is "...the fluid of choice on contemplated Space Station Freedom thermal control systems." (7, p. 10)

References/Applicable Publications:
(1) Ku, J., et al.: Capillary Pumped Loop Gas and Hitchhiker Flight Experiments. Fourth AIAA and ASME Joint Thermophysics and Heat Transfer Conference, Boston, Massachusetts, June 2-4, 1986, 15 pp., AIAA Paper #86-1249. (post-flight; Note: this document does not discuss the STS-023 experiments.)

(2) Butler, D.: The Capillary Pumped Loop (CPL) Gas Experiment G-471. In Goddard Space Flight Center's 1985 Get Away Special Experimenter's Symposium, October 8-9, 1985, NASA CP-2401, pp. 237-253. (post-flight)

(3) Cargo Systems Manual: Gas Annex For STS 51-D, JSC-17645 51-D, March 20, 1985. (short description; preflight)

(4) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, the NASA GAS Team 1989, p. 25. (post-flight, very brief description)

(5) NASA STS 51-D Press Kit, April 1985. (preflight)

(6) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(7) Hill, M. E. and O'Malley, T. F.: A Summary of Existing and Planned Experiment Hardware for Low-Gravity Fluids Research. AIAA 29th Aerospace Sciences Meeting, January 7-10, 1991, Reno, Nevada, AIAA-91-0777, pp. 9-10 (also NASA TM-103706). (post-flight)

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Experiment Origin: USA
Mission: STS Launch #18, STS-025 (STS 51-G, Discovery)
Launch Date/Expt. Date: June 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) canister G-471R
 Volume of Canister: 5.0 cubic feet
 Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-471R: NASA Goddard Space Flight Center, Greenbelt, Maryland/OAO, Inc., Greenbelt, Maryland
Processing Facility: A miniaturized capillary pumped loop heat pipe system which included (1) two capillary pumped evaporators mounted in parallel, (2) integral heaters attached directly to the outer surfaces of the evaporators, (3) a fluid loop, and (4) a condenser plate.
Builder of Processing Facility: The OAO Corporation, Greenbelt, Maryland, built the miniaturized capillary pumped loops.

Experiment:
Capillary Pumped Loop (CPL)

This experiment was the second in a series of investigations designed by McIntosh et al. to demonstrate the capability of a capillary pumped loop (CPL) system (operating in low gravity) to achieve thermal control (see McIntosh, STS-023). (A detailed description of the CPL heating and cooling cycle can be found under McIntosh, STS-023).

The STS-025 experiment was nearly identical to the earlier STS-023 CPL investigation. In fact, the experimental hardware and anticipated experiment sequence appears to have been the same. The only significant change mentioned for the STS-025 payload was related to a battery failure that occurred on STS-023. Because it was thought that the STS-023 GAS batteries failed to operate in the vacuum and cold temperatures of space, the STS-025 batteries were enclosed in a hermetically sealed box.

As earlier detailed under McIntosh, STS-023, the experiment was developed using a significant amount of hardware which had flown in support of previous (essentially unrelated) GAS experiments. Thus, the size of the CPL system which could be examined was somewhat limited by the volume requirements of this available hardware. Nonetheless, a "mini-CPL" system, which maintained

most of the operating features of the ground-based system was developed.

The mini-CPL system, which measured approximately 14" long by 14" wide by 4" high, was mounted directly to the GAS top plate. The system included (1) two capillary pumped evaporators mounted in parallel to a common vapor header, (2) integral heaters attached directly to the outer surfaces of the evaporators (simulating the heat dissipation from a spacecraft component), (3) a single multi-pass condenser tube attached to a condenser plate (heat sink), (4) a temperature-controlled, two-phase reservoir, (5) a subcooled liquid return line, and (6) various control electronics. The "...15 pound condenser plate and the 25 pound GAS top plate to which the condenser plate... [was] mounted... [comprised] the system heat sink. (1, p. 3)

The importance of the two-phase reservoir was introduced under McIntosh, STS-023. A further discussion of the reservoir was reported in Reference (1). "One of the... [principle] issues that had to be addressed in the flight experiment was the design of a two-phase reservoir for zero-g application. Breadboard and engineering model prototypes tested previously had utilized gravity to ensure only liquid entered the CPL loops from their reservoirs. To obtain the same result in the zero-g environment, the reservoir line was connected to the condenser plate before reaching the CPL loop. The... [intended] purpose was to condense any vapor leaving the reservoir. This feature was necessary in order to avoid a deprime of the CPL system as a result of vapor displacement. Also, reservoir liquid/vapor management in zero-g required capillary devices to maintain liquid/vapor separation and to ensure preferential liquid displacement. Consequently, a reservoir design that employed wicked liquid acquisition baffles was developed for the GAS/CPL flight experiment." (1, p. 3)

The experiment operating times were based on a (preflight) thermal control analysis of the CPL GAS payload. This analysis indicated that the payload could accommodate "...experiment heater cycles of up to 220 watts total (110 watts on each pump) for operating times of up to one hour, followed by a cool down period lasting approximately 10 hours." (2, p. 241)

<Note: Reference (1) reported different heater loads: "...the planned heater cycles included a maximum power input of up to 200 watts total (100 watts per evaporator) for operating periods of up to one hour, followed by cooldown periods lasting about nine hours." (1, p. 3)>

Reportedly, the expected heating/cooling sequence of the payload during a typical planned heating cycle was to include the following: "The experiment condenser was... [to initially] cool to approximately 5 ^{O}C, then the experiment heaters were [to be] activated. Since the power input... [was expected to exceed] the instantaneous heat rejection capability of the GAS top plate, the condenser temperature... [would increase]. When the condenser temperature... [approached] the CPL operating temperature of 29 ^{O}C it... [would no longer be able to] absorb any more heat and the system... [would] shut down and... cool down. The heater cycle... [would then be] repeated after the condenser... [cooled] back down to about 5 ^{O}C. During the flight, the cycles [were to be] repeated for the total mission time, approximately 120 hours." (2, p. 241) (Other cycle scenarios were also planned.)

Reportedly, "A total of 13 power profiles were run during the 120 hour mission. The majority of the power cycles involved heating each evaporator with a constant 100 watts for 30 minutes; other profiles were interspersed.... The definitions and objectives of the cycles are described below:

"a. ...Heat input of 100 watts was applied to each evaporator. The purpose was to verify that the same start-up procedure used... [on] the ground could be applied in the zero-g environment with similar results.

"b. ...Heat input of 100 watts was applied to one evaporator only. The objective of this test was to demonstrate that vapor from the heated evaporator would backflow into the other and condense there. This showed that the CPL can not only be used to transfer heat away from a heat source, but also to transport heat to unheated evaporators.

"c. ...Heat input of 25 watts was applied to each evaporator. The purpose of this test was to find out whether the system would prime under such a low power condition in the zero-g environment.

"d. ...Initial heat input of 100 watts was applied to each evaporator followed by heat input to one of the evaporator inlets to intentionally deprime that evaporator pump. The objective was to demonstrate the ability of the CPL system to isolate a single pump after it has deprimed so that the remainder of the system would continue to function normally.

"e. ...Initial heat input of 100 watts was applied to each evaporator followed by a reduction of power to one of the evaporators. The purpose was to demonstrate the ability of the system to adjust to changes in the system power input." (1, p. 4)

Post-flight analysis of the payload indicated that the experiment was "very successful" and that (1) the GAS relays operated satisfactorily, (2) the mini-CPL operated for the planned 120 hours (demonstrating the 13 power cycles), (3) all of the power profiles could be examined (even the low power cycle which would not work on the ground), and (4) none of the cycles deprimed.

<Note: The low power cycle referred to above probably pertains to an operational profile proposed by OAO Corporation which was to begin "...with lower power on the pumps (25 watts each), with 25 watt step increases to 100 watts each at the end of 45 minutes. When this power profile was tried [on the ground] the evaporator pumps deprimed soon after startup and would no longer carry the applied heat load, as evidenced by a sudden rise in their temperature." (2, p. 242)>

Reference (5) reported that "Little difference was found between normal-gravity and microgravity system performance." (5, p. 10)

Because the majority of the power profiles were that of cycle (1) detailed above, an abridged discussion of the results pertaining to this cycle are presented:

"The reservoir temperature controller was set at 29 $^{\circ}$C during this cycle as it was for the full 120-hour mission. With the evaporators at 7 $^{\circ}$C, power was applied, and the evaporator temperatures rose quickly to reach the system saturation temperature as determined by the reservoir set point. Consequently, liquid ammonia began to vaporize, and the pumping action inside the evaporators started. The evaporator temperatures remained steady at 30 $^{\circ}$C until the heater power was turned off about thirty minutes later...." (1, p. 4) Other details of the cycle can be found in Reference (1).

A discussion of the results from some of the other power cycles were also presented in Reference (1).

Key Words: Technological Experiments, Heat Pipes, Thermal Control, Heat Transfer, Vaporization, Evaporators, Evaporation, Condensers, Condensation, Phase Transition, Phase Separation, Wicking, Baffles, Surface Tension, Capillary Flow, Capillary Forces, Liquid Transfer, Liquid/Vapor Interface, Fluid Management

Number of Samples: one mini CPL system
Sample Materials: The working fluid was ammonia, NH_3.
(N*H*)
Container Materials: unknown

Experiment/Material Applications:
See McIntosh, STS-023.

References/Applicable Publications:

(1) Ku, J., Kroliczek, E. J., Butler, D., Schweickart, R. B., and McIntosh, R.: Capillary Pumped Loop Gas and Hitchhiker Flight Experiments. Fourth AIAA and ASME Joint Thermophysics and Heat Transfer Conference, Boston, Massachusetts, June 2-4, 1986, 15 pp., AIAA Paper #86-1249. (post-flight)

(2) Butler, D.: The Capillary Pumped Loop (CPL) GAS Experiment G-471. In 1985 Get Away Special Experimenter's Symposium, NASA CP-2401, NASA Goddard Space Flight Center, Greenbelt, Maryland, October 8, 1985, pp. 237-253. (post-flight)

(3) Kolcum, E. H.: Fuel Contaminant Threatens Delay in Shuttle Launch. Aviation Week and Space Technology, June 17, 1985, p. 21. (preflight)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(5) Hill, M. E. and O'Malley, T. F.: A Summary of Existing and Planned Experiment Hardware for Low-Gravity Fluids Research. AIAA 29th Aerospace Sciences Meeting, January 7-10, 1991, Reno, Nevada, AIAA Paper #91-0777, pp. 9-10; also NASA TM-102706. (post-flight)

(6) NASA STS 51-G Press Kit, June 1985, p. 21. (preflight)

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Experiment Origin: USA
Mission: STS Launch #24, STS-032 (STS 61-C, Columbia)
Launch Date/Expt. Date: January 1986
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Hitchhiker HHG-01-B (A Get Away Special (GAS) canister modified to be installed on the GSFC Hitchhiker system.)
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of HHG-01: NASA Goddard Space Flight Center, Greenbelt, Maryland
Processing Facility: A miniaturized capillary pumped loop heat pipe system which included (1) two capillary pumped evaporators mounted in parallel, (2) integral heaters attached directly to the outer surfaces of the evaporators, (3) a fluid loop, and (4) a condenser plate
Builder of Processing Facility: The OAO Corporation, Greenbelt, Maryland, built the miniaturized capillary pumped loops.

Experiment:
Capillary Pump Loop Experiment (CPL)

This experiment was the third in a series of investigations designed by McIntosh et al. to demonstrate the capability of a capillary pumped loop (CPL) system to achieve thermal control (see McIntosh, STS-023; STS-025). (A detailed description of the CPL heating and cooling cycle can be found under McIntosh, STS-023.) The specific objective of this Hitchhiker experiment was to acquire CPL performance data at power levels higher than those levels which had been available on STS-025.

During the earlier STS-025 Get Away Special (GAS) experiment, a battery contained within the GAS canister provided a maximum power input of approximately 200 watts (100 watts per evaporator) to a mini-CPL system. In contrast, this STS-032 experiment (which was carried in a modified GAS canister known as Hitchhiker) was now powered by a space shuttle source. Thus, the maximum power input to each evaporator could be increased to approximately 400 watts.

The STS-032 CPL system was very similar to the earlier STS-025 CPL investigation. The only significant hardware changes mentioned for the STS-032 payload were related to the accommodation of increased heat loads. "The mass of the GAS container top plate was increased from 25 to 140 pounds to increase the con-

denser heat sink capability. Additional modifications included removal of the thermal insulation blankets from the sides of the gas container. This change increased the effective radiating area of the container by a factor of three and, thus, enhanced its heat rejection capability." (1, p. 6)

Because no further mention was made of modifications to the CPL system, it was assumed that the configuration was essentially the same as that on STS-025. Thus, the mini-CPL system, which was mounted directly to the canister top plate, included (1) two capillary pumped evaporators mounted in parallel to a common vapor header, (2) integral heaters attached directly to the outer surfaces of the evaporators (simulating the heat dissipation from a spacecraft component), (3) a single multi-pass condenser tube attached to a condenser plate (heat sink), (4) a temperature-controlled, two-phase reservoir, (5) a subcooled liquid return line, and (6) various control electronics. The importance of the two-phase reservoir was introduced under McIntosh, STS-023, and is further discussed under McIntosh, STS-025.

"The power cycles for the CPL on the Hitchhiker-G carrier were similar to that for [STS-025] CPL/GAS. For most cycles, the system was allowed to cool to 5 ^{O}C or below before power was applied. Again, even with the improved heat rejection capability, the CPL... radiator could not reject as much heat as the evaporator heaters could supply. Thus, when the condenser heat sink capacity was exhausted, the heaters were shut off, and the system was allowed to cool. A measured improvement was evident, though since the heat rejection capacity of the [STS-032] CPL... allowed for power cycles up to 600 watts for 40 minutes followed by a four hour cool-down compared to 200 watts for 30 minutes with a nine hour cool-down cycle for the [the STS-025] CPL/GAS configuration." (1, p. 6)

Reportedly, a total of 38 power profiles were run during the 5-day mission. The definitions and objectives of the cycles are described below:

"a. ...Various constant power levels were applied to both evaporators. The objective of these tests were to verify the system start-up and operation at power levels that spanned the CPL's operating range.

"b. ...Power was applied to both evaporators, with subsequent increases or decreases in power to both. The purpose of these tests were to find the high power system limit and to demonstrate the ability of the system to adjust to an abrupt change in heat loads.

"c. ...After the heat sharing mode of operation was confirmed, power was applied to the unheated evaporator. The purpose of this test was to demonstrate the ability of the evaporator to prime with an inlet temperature level near the saturation temperature. Also shown was the ability of an individual pump to convert from evaporator mode to condenser mode and vise versa without any active external control measures.

"d. ...The reservoir set point was changed or allowed to vary. The objective of this test was to demonstrate that the system saturation temperature could be varied via the reservoir controller while the system continued to operate normally.

"e. ...Power was applied to an evaporator and was subsequently increased to force an evaporator dry-out. Power to the dried out evaporator was then shut off and the evaporator was allowed to cool. When the evaporator temperature dropped below the saturation temperature, power was re-applied. The purpose of this test was to demonstrate quick recovery of an evaporator from a dry-out condition.

"f. ...Power levels below 100 watts per evaporator were applied to each evaporator. The objective of these tests were to investigate the deprime phenomenon at lower power levels.

"g. ...Power was applied to both evaporators when the condenser temperature was just below the saturation temperature. The objective of this cycle was to determine the minimum required temperature differential between the reservoir and the evaporator inlets that would still permit proper system operation." (1, p. 5-6)

Post-flight analysis of the payload indicated that the experiment was "very successful" and that (1) high and low power limits for the Hitchhiker CPL were determined and (2) proper system operation in low gravity was verified. Further, because the Hitchhiker system (1) allows data to be examined in real time on the ground and (2) recognizes real-time commands from the ground, "...real-time changes of the power profiles were accomplished, and therefore, maximum, utilization of the experiment was realized." (1, p. 6)

It was reported that "...no significant differences between zero-g and one-g performance of the CPL have been identified to date. This has increased confidence that CPL system performance can be verified on the ground prior to implementation in space. Nonetheless, further zero-g CPL experimentation is required on larger systems to investigate zero-g effects on long transport distances, new condenser configurations and scaling laws.

"A larger CPL consisting of four evaporators and four parallel condensers with 10-meter transport lengths will be flown... in the future." (1, p. 8)

A discussion of the results from many of the power cycles were presented in Reference (1).

Key Words: Technological Experiments, Heat Pipes, Thermal Control, Heat Transfer, Vaporization, Evaporators, Evaporation, Condensers, Condensation, Heat Radiators, Phase Transition, Phase Separation, Wicking, Surface Tension, Capillary Flow, Capillary Forces, Liquid Transfer, Liquid/Vapor Interface, Fluid Management

Number of Samples: one CPL system
Sample Materials: The working fluid was ammonia, NH_3.
(N*H*)
Container Materials: unknown

Experiment/Material Applications:
See McIntosh, STS-023.

References/Applicable Publications:

(1) Ku, J., Kroliczek, E. J., Butler, D., Schweickart, R. B., and McIntosh, R.: Capillary Pumped Loop GAS and Hitchhiker Flight Experiments. Fourth AIAA/ASME Joint Thermophysics and Heat Transfer Conference, June 2, 1986, AIAA Paper #86-1249. (post-flight)

(2) Modified Columbia will Deploy RCA Satellite on Next Mission. AW&ST, December 16, 1985. (concerns Hitchhiker facility only; preflight)

(3) Mordoff, K. F.: Commercialization of Space: Shuttle Hitchhiker Spurs Business Effort. AW&ST, June 25, 1984. (concerns Hitchhiker facility only; preflight)

(4) Grote, M. G., Stark, J. A., Butler, C. D., and McIntosh, R.: Design and Test of a Mechanically Pumped Two-Phase Thermal Control Flight Experiment. AIAA 22nd Thermophysics Conference, June 8-10, 1987, Honolulu Hawaii, AIAA Paper #87-1629. (future payload)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Company, Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

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Experiment Origin: USA
Mission: STS Launch #3, STS-3 (STS OFT-3, Columbia)
Launch Date/Expt Date: March 1982
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Middeck Experiment, Crew Compartment
Processing Facility: Monodisperse Latex Reactor (MLR): four one-foot-tall chemical reactors, each containing 100 milliliters of a chemical latex forming mixture, housed in a single, two-foot tall Experiment Apparatus Container (EAC, metal cylinder); reaction time: 12 hours
Builder of Processing Facility: Reactors: General Electric Co., Valley Forge, Pennsylvania; Support Electronics Package (SEP): Rockwell International, Downey, California

Experiment:
<u>Monodisperse Latex Reactor (MLR-1)</u>

Latex is a suspension of small plastic spheres, usually in water. Larger monodisperse (identical size) latex particles are produced by adding monomers, initiators, and emulsifiers to a monodisperse latex of a relatively small particle size. The monomer, which in this case is totally soluble in the microspheres, is absorbed into the existing "seed" microspheres; the seeds all swell equally to the desired size. Upon heating, the initiator starts polymerization of the monomer absorbed within the spheres, and the emulsifiers help prevent coagulation. Thus larger particles are grown from smaller seed particles, hopefully without (1) generating coagulum, or (2) nucleating a new crop of smaller particles.

On Earth, the largest monodisperse latex particles which can be readily produced have diameters of several micrometers. Generally the larger the particles produced, the worse their monodispersity. Monodisperse latex particles can be grown by emulsion, suspension, or dispersion polymerization techniques but during the MLR program, only emulsion polymerization "recipes" were flown. In this process, once the high density polymer seed particles have been swollen with low-density monomer, the average density of the particles becomes less than 1, and the particles tend to "cream" or float (in the Earth system) to the top of the

container. After polymerization begins, the average density of the growing particles continues to increase as the absorbed monomer is being converted to polymer, until particle density has again reached that of pure polymer. The larger these new particles are, the faster they settle. A stirring process can prevent creaming and settling. However, the stirring required to maintain the suspension causes particles to collide and stick together. Coagulation of the particles results because of the shear-force sensitive nature of the particles. Since buoyancy is significantly reduced under low-gravity conditions, the creaming and settling is eliminated and the multi-density suspension is maintained.

This STS-003 experiment was the first in a series of investigations designed by Vanderhoff et al. to study the low-gravity production of monodisperse polystyrene latex microspheres.

An apparatus known as the Monodisperse Latex Reactor (MLR) was used to produce the latex particles. The flight apparatus consisted of (1) four reactor cylinders (0.3 meter tall) contained within an Experiment Apparatus Container (EAC), and (2) the Support Electronics Package (SEP). Each stainless steel reactor cylinder (dilatometer) contained a stirring device for gentle agitation (to insure uniform temperature) and a piston to indicate the volume change during the reaction. (The polymerization conversion-time curves were deduced from the decrease in volume.) Each dilatometer contained four, three-pellet diodes for temperature measurement. The diodes were located (1) in a probe extending into the center of the chamber, (2) at the top interior surface, (3) in the chamber wall midway between top and bottom, and (4) at the bottom of the chamber, next to the stirrer shaft. Heat was supplied to the dilatometers by a heating tape wrapped around the outside of each reactor. Experiment control was provided by microprocessors mounted on each of the four reactor cylinders. The SEP provided the proper regulated dc voltage to the reactors, and contained (1) a data tape recorder and (2) a malfunction detection system.

Four different particle seed sizes (and their accompanying monomer-polymer ratios) were selected for the STS investigation: latex number 1 contained seeds of 2.5 microns and had a monomer-polymer ratio of 2:1; latex number 2 contained seeds of 2.5 microns and had a monomer-polymer ratio of 4:1; latex number 3 contained seeds of 2.5 microns and had a monomer-polymer ratio of 10:1; latex number 4 contained seeds of 0.19 microns and had a monomer-polymer ratio of 2:1. The 0.19 micron sized seed experiment used a potassium persulfate initiator and a sodium bicarbonate buffer (see References (2), (8), (9), or (10) for other latex recipe details).

Prior to the mission, an exact amount of (1) styrene monomer, (2) azo initiator, (3) inhibitor, and (4) emulsifier was added to the seed latex under vacuum and at room temperature. After adding these materials to each of the four latex reactors and then sealing the reactors, constant stirring began in each of the cylinders (13 rpm) and continued (1) during payload integration, (2) vehicle launch, (3) prior to and until the on-orbit processing steps had begun, and (4) throughout the duration of the experiment. At the beginning of the low-gravity processing procedure, each mixture was heated to 70 ^{o}C and, if required, the stirrer speed was automatically adjusted. (Since particle size consistency depends on the isothermal nature of the latex batch (a temperature variation within different areas of the chamber of even 1 ^{o}C will significantly increase the growth rate of the polymer within the hot region), the material was gently stirred to prevent temperature differences within the batch material.) Once the particles became hot, the added monomer (which had been absorbed by the seed particles and caused them to swell) polymerized resulting in a slight particle shrinkage. This shrinkage was measured, recorded on the data tape recorder in the SEP, and used to calculate the reaction rates. After 10.4 hours at 70 ^{o}C, the temperature was increased to 90 ^{o}C for 0.75 hours to destroy the remaining traces of the initiator and push the reaction to 100% completion. After the mission, the dilatometers were cleaned and ground control experiments were performed.

Post-flight examination of the latex particles and recorded data indicated that the experiment using the 0.19 micron sized seed particles polymerized prematurely and, therefore, no reaction rate data were available for this experiment. However, each of the remaining three experiments (2.5 micron seed) produced a very good latex product. All latexes were completely polymerized and were monodisperse. Latex number 2 did contain a small lump of coagulum which restricted the motion of the stirrer after the flight. However, the remaining latexes contained negligible amounts of coagulum. The reported product size for (1) latex number 1 was 3.4 microns, (2) latex number 2 was 4.1 microns, and (3) latex number 3 was 5.0 microns. It was reported that "...there were subtle differences in particle size distribution between the three flight latexes and the ground-based control latexes. The coefficients of variation were about the same for all latexes except for ground-based control latex 3, which was broader in particle size distribution. The standard deviations increased only slightly with increasing particle size." (10, p. 3)

The conversion-time curves for the flight polymerizations were essentially the same as the curves of the ground-based polymerizations. The 2:1 monomer to polymer ratio had a significant upward deviation from linearity indicating autoaccelera-

tion. <Note: Autoacceleration was defined as "...when the viscosity of the medium (inside the spheres) increases to the point where the rate of termination decreases, and at the same time the rate of propagation remains constant, so that the net overall effect is an increase in the overall rate of polymerization. During this period of autoacceleration (maximum polymerization rate) the particles become especially susceptible to shear-induced coagulation." (11)> The 4:1 ratio had only a slight upward deviation, and the 10:1 ratio had a nearly linear variation. "Since the critical particle size for the transition from emulsion polymerization kinetics to bulk polymerization kinetics is ca. 1.3... [microns] for the styrene-polystyrene system at 70... [degrees] [see Reference (10)], the polymerization rate should be proportional to the monomer concentration and the square root of the initiator concentration in the absence of autoacceleration. The upward deviation from linearity began earlier, the lower the monomer-polymer ratio, as expected from the higher viscosity of the particles." (10, p. 5)

Key Words: Technological Experiments, Emulsion Polymerization, Monodisperse Latex Particles, Polystyrene Latex Microspheres, Spheres, Suspension of Particles, Stirring of Components, Shear Forces, Sedimentation, Nucleation, Particle Growth, Particle Size Distribution, Collisions, Coagulation, Buoyancy Effects Diminished, Thermal Control, Isothermal Processing, Piston System, Volume Change, Volume Compensation, Contained Fluids, Viscosity, Vacuum, Processing Difficulties

Number of Samples: four
Sample Materials: Monomer: styrene, water; Latex #1: seed diameter = 2.5 microns at 2/1 monomer/polymer ratio; Latex #2: seed diameter = 2.5 microns at 4/1 monomer/polymer ratio; Latex #3: seed diameter = 2.5 microns at 10/1 monomer/polymer ratio; Latex #4: seed diameter 0.19 microns at 2/1 monomer/polymer ratio; buffer (Latex 4 only): sodium bicarbonate; initiator (Latex 4 only): potassium persulfate (see References (8), (9), or (10) for other recipe details)
Container Materials: stainless steel

Experiment/Material Applications:
"There are many uses for monodisperse latex particles. Medical applications include pore size standards for stomach, peritoneal cavity, membrane and intestinal wall pores for ongoing cancer research, eye exit channel pore size for glaucoma research, and do-it-yourself tests. Research to develop carriers for drugs and radioactive isotopes inside tumors and organs that will control dosages, increase drug effectiveness, and reduce toxicity is being done. As calibration standards, the particles will be used in blood cell counters, as internal standards in electron and optical microscopes, and in filter calibration. Chemical analysis of biological and other material will be enhanced through chromatography column packing. Anti-blocking agents for the plastics industry is another area in which the monodisperse latex will be used...." (6, p. 8)

References/Applicable Publications:
(1) Tseng, C.-M., El-Aasser, M. S., and Vanderhoff, J. W.: Modeling the Equilibrium Swelling of Latex Particles. In Computer Applications in Applied Polymer Science, edited by T. Provder, ACS Symp. Ser. 197, pp. 197-208 (1982).

(2) Kornfeld, D. M.: Monodisperse Latex Reactor (MLR) - A Materials Processing Space Shuttle Middeck Payload. NASA TM-86487, January 1985. (post-flight)

(3) STS 3rd Space Shuttle Mission, March 1982, NASA Press Kit, pp. 63-64. (preflight)

(4) STS-7 Seventh Space Shuttle Mission. June 1983, NASA Press Kit, p. 51. (briefly discusses success of STS-003)

(5) "First Space Product Set to be Developed for Commercial Use." NASA Activities, August 1984, Vol. 15, No. 8, pp. 5-6. (post-flight)

(6) Monodisperse Latex Reactor (MLR). Application Payload Projects, Spacelab Payload Project Office, NASA Marshall Space Flight Center, Huntsville, Alabama, 8 pp. (document prepared by Teledyne Brown Engineering)

(7) Vanderhoff, J. W., El-Aasser, M. S., Micale, F. J., Sudol, E. D., Tseng, C.-M., Silwanowicz, A., and Kornfeld, D. M.: Preparation of Large-Particle Size Monodisperse Latexes in Space. J. Dispersion Sci. Technology, Vol. 5, pp. 231-246, 1984.

(8) Vanderhoff, J. W., El-Aasser, M. S., Micale, F. J., Sudol, E. D., Tseng, C.-M., Silwanowicz, A., and Kornfeld, D. M.: Preparation of Large-Particle-Size Monodisperse Latexes in Space. Polm. Materials Sci. Eng., Vol. 54, pp. 587-592, 1986.

(9) Vanderhoff, J. W., El-Aasser, M. S., Kornfeld, D. M., Micale, F. J., Sudol, E. D., Tseng, C.-M., and Sheu, H.-R.: The First Products Made in Space: Monodisperse Latex Particles. In Materials Processing in the Reduced Gravity Environment of Space, Fall Meeting, Materials Research Society, December 1-6, 1986, Boston, Massachusetts, Mat. Res. Soc. Symp. Proc., Vol. 87, 1987.

(10) Vanderhoff, J. W., El-Aasser, M. S., Micale, F. J., Sudol, E. D., Tseng, C.-M., Sheu, H.-R., and Kornfeld, D. M.: The First Products Made in Space: Monodisperse Latex Particles. AIAA 25th Aerospace Sciences Meeting, January 12-15, 1987, Reno, Nevada, AIAA Paper #87-0389.

(11) Input received from Co-Investigator D. Kornfeld, September 1989 and July 1993.

For additional publications, see Vanderhoff, STS-004 (publications listing).

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Experiment Origin: USA
Mission: STS Launch #4, STS-004 (STS OFT-4, Columbia)
Launch Date/Expt Date: June 1982
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Middeck Experiment, Crew Compartment
Processing Facility: Monodisperse Latex Reactor (MLR): four one-foot-tall chemical reactors, each containing 100 milliliters of a chemical latex forming mixture, housed in a single, two-foot tall Experiment Apparatus Container (EAC, metal cylinder); reaction time: 12 hours
Builder of Processing Facility: Reactors: General Electric Co., Valley Forge, Pennsylvania; Support Electronics Package (SEP): Rockwell International, Downey, California

Experiment:
Monodisperse Latex Reactor (MLR-2)

This STS-004 experiment was the second in a series of investigations designed by Vanderhoff et al. to study the low-gravity production of monodisperse polystyrene latex microspheres (see Vanderhoff, STS-003).

The Monodisperse Latex Reactor (MLR), which had been previously employed on STS-003 to produce latex particles, was reused during this STS-004 mission. The STS-004 latex production experimental procedure was essentially the same as that of the earlier STS-003 mission (see Vanderhoff, STS-003 for details of the MLR and a description of the procedure).

During the STS-004 experiment, four space-based polymerizations (designated latex number 5, number 6, number 7, and number 8) were carried out using seeds of 5.5 microns. The monomer-polymer ratios for flight latexes 5, 6, 7, and 8 were 2:1, 4:1, 5.7:1, and 6.2:1, respectively. The pre-processing and processing stirring rates (both 13 rpm) were the same for all four experiments.

Post-flight examination of (1) the returned liquid mixtures and (2) the recorded experimental data revealed that "All four latexes were incompletely polymerized as evidenced by the odor of styrene; moreover, the data tape cassette yielded only meaning-

less numbers for the dilatometer volume and temperature readings. A dc voltage converter in the SEP had failed, with the consequent failure of other electronic components, so that the temperature time variation of the monomer-swollen latexes was not known and the voltage signals to the data tape cassette were inconsistent and non-representative." (12, p. 5)

The degree of conversion of the partially polymerized latexes as determined by gravimetric measurements was reported to be 48%-67%; the degree of conversion as determined by ultraviolet absorbance of isooctane was 54%-73%. Optical microscopy indicated the particles were monodisperse with only a few offsize (larger) particles. The size of the particles was as expected from the stoichiometry of the seeded polymerizations: (1) 7.2 microns for the 2:1 monomer-polymer ratio, (2) 8.6 microns for the 4:1 ratio, (3) 9.5 microns for the 6:1 ratio, and (4) 10.4 microns for the 8:1 ratio. The residual monomer within the latex particles rendered them useless as calibration standards. "Moreover, completion of the polymerizations on Earth gave a broader particle size distribution and an increased number of larger offsize particles, the result of further coalescence of e monomer-swollen particles during polymerization." (12, p. 5) <Note: The Principal Investigator deliberately completed the polymerization on Earth by heating the four samples to 70 ^{o}C in order to determine the degree of monodispersity.>

Key Words: Technological Experiments, Emulsion Polymerization, Monodisperse Latex Particles, Polystyrene Latex Microspheres, Spheres, Suspension of Particles, Stirring of Components, Shear Forces, Nucleation, Particle Growth, Particle Size Distribution, Sedimentation, Isothermal Processing, Buoyancy Effects Diminished, Thermal Control, Contained Fluids, Piston System, Vacuum, Incomplete Sample Processing, Hardware Malfunction, Processing Difficulties

Number of Samples: four
Sample Materials: Latex #5: seed diameter = 5.5 microns at 2/1 monomer/polymer ratio; Latex #6: seed diameter = 5.5 microns at 4/1 monomer/polymer ratio; Latex #7: seed diameter = 5.5 microns at 5.7/1 monomer/polymer ratio; Latex #8: seed diameter 5.5 microns at 6.2/1 monomer/polymer ratio
Container Materials: stainless steel

Experiment/Material Applications:
See Vanderhoff, STS-003

References/Applicable Publications:

(1) Sudol, E. D., Micale, F. J., El-Aasser, M. S., and Vanderhoff, J. W.: The Development and Testing of a Space Flight Dilatometer/Reactor. Rev. Sci. Instruments Vol. 57, pp. 2332-2338 (1986).

(2) Input received from Co-Investigator D. Kornfeld, September 1989 and July 1993.

(3) Kornfeld, D. M.: Monodisperse Latex Reactor (MLR) - A Materials Processing Space Shuttle Middeck Payload. NASA TM-86487, January 1985. (post-flight)

(4) STS-4 Fourth Space Shuttle Mission, June 1982, NASA Press Kit, pp. 50-52. (preflight)

(5) "Applications Experiments on Columbia." In NASA Mission Report, STS-4, MR-004. (post-flight; very short summary)

(6) STS-6 Sixth Space Shuttle Mission, April 1983, NASA Press Kit, p. 38. (briefly mentions hardware failure during STS-4.)

(7) STS-7 Seventh Space Shuttle Mission, June 1983, NASA Press Kit, pp. 51-52. (briefly mentions hardware failure during STS-4.)

(8) "First Space Product Set to be Developed for Commercial Use." NASA Activities, August 1984, Vol. 15, No. 8, pp. 5-6. (post-flight)

(9) Monodisperse Latex Reactor (MLR). Application Payload Projects, Spacelab Payload Project Office, Marshall Space Flight Center, Huntsville, Alabama, 8 pp. (document developed by Teledyne Brown Engineering)

(10) Sudol, E. D., El-Aasser, M. S., and Vanderhoff, J. W.: Kinetics of Successive Seeding of Monodisperse Polystyrene Latexes I. Initiation via Potassium Persulfate. Accepted for publication in J. Polym. Sci., 1986. <Note: The current status of this document is unclear at this time.>

(11) Sudol, E. D., El-Aasser, M. S., and Vanderhoff, J. W.: Kinetics of Successive Seeding of Monodisperse Polystyrene Latexes II. Azo Initiators with and without Inhibitors. Accepted for publication in J. Polym. Sci., 1986. <Note: The current status of this document is unclear at this time.>

(12) Vanderhoff, J. W., El-Aasser, M. S., Micale, F. J., Sudol, E. D., Tseng, C.-M., Sheu, H.-R., and Kornfeld, D. M.: The First Products Made in Space: Monodisperse Latex Particles. AIAA 25th Aerospace Sciences Meeting, January 12-15, 1987, Reno, Nevada, AIAA Paper #87-0389.

For additional publications see Vanderhoff, STS-003.

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Experiment Origin: USA
Mission: STS Launch #6, STS-006 (STS 31-B, Challenger)
Launch Date/Expt Date: April 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Middeck Experiment, Crew Compartment
Processing Facility: Monodisperse Latex Reactor (MLR) (same apparatus as employed on STS-003 and STS-004, but now with (1) a cooling fan installed in the Support Electronics Package (SEP), (2) some electronic modifications, and (3) a software change to increase processing to 20 hours.
Builder of Processing Facility: Reactors: General Electric Company, Valley Forge, Pennsylvania; Support Electronics Package (SEP): Rockwell International, Downey, California

Experiment:
Monodisperse Latex Reactor (MLR-3)

This STS-006 experiment was the third in a series of investigations designed by Vanderhoff et al. to study the low-gravity production of monodisperse polystyrene latex microspheres (see Vanderhoff, STS-003, STS-004).

The Monodisperse Latex Reactor (MLR), which had been previously employed on both STS-003 and STS-004 to produce latex particles, was reused during this STS-006 mission. The STS-006 latex production experimental procedure was essentially the same as that of the earlier STS-003 mission (see Vanderhoff, STS-003).

Specifically, during the STS-006 experiment, four space-based polymerization experiments were conducted using different seed sizes and monomer-polymer ratios: latex number 9 used 5.6 micron seeds and a monomer/polymer ratio of 2:1; latex number 10 used 5.6 micron seeds at a monomer/polymer ratio of 4:1; latex number 11 used 5.6 micron seeds at a monomer/polymer ratio of 6:1; latex number 12 used 0.19 micron seeds at a monomer/polymer ratio of 2:1. (Latex number 12 was a recipe repeat of latex number 4 which was processed during the STS-003 experiment. Further, this 0.19 micron sized seed experiment used a potassium persulfate initiator and a sodium bicarbonate buffer (see Reference (9) for recipe details).) The pre-processing and processing stirring

speeds (both 13 rpm) were the same for all four experiments.

Post-flight examination of the returned liquid mixtures revealed that the "Flight latex 10 displayed a strong odor of styrene; this sample had not polymerized owing to a broken wire in the heating circuit. It is not known whether the wire broke before or during the launch; however, the reactor functioned satisfactorily in the ground-based test polymerizations carried out two weeks before the flight." (9, p. 5)

Examination of the remaining flight samples indicated a smaller variation in size than the ground-based samples. Latex number 9 contained 7.9 micron diameter spheres, latex number 11 contained 10.0 micron diameter spheres, and latex number 12 spheres contained 0.26 micron diameter spheres. Both flight latexes 9 and 11 were more uniform in size than the corresponding ground-based samples. "Flight latex 11 (9.96... [microns] diameter) was accepted by the National Bureau of Standards as a Standard Reference Material and went on sale in July 1985, the first product made in space for sale on earth. These particles were also found to be more perfect spheres than the ground-based particles." (9, p. 5)

The conversion-time curves were similar for both flight and ground-based latexes (this similarity was also indicated for the STS-003 flight and ground-based latexes). The curves for latex number 9 (both ground-based and flight) demonstrated a significant upward deviation from linearity to non-linearity, indicative of autoacceleration. Similar curves for latexes 11 showed nearly linear variations. "The failure of the curves for latexes 11 to show an upward deviation from linearity was attributed to the sticking of the dilatometers or formation of nitrogen bubbles before the polymerizations reached the autoacceleration stage." (9, p. 6) (Reportedly, decomposition of the azo initiator liberates nitrogen gas; excess gas can form a bubble in the fluid chamber. Since bubbles are compressible and water is not, the employed volume compensation piston may not move properly.) <Note: A general discussion on conversion-time curves can be found under Vanderhoff, STS-003.>

Key Words: Technological Experiments, Emulsion Polymerization, Monodisperse Latex Particles, Polystyrene Latex Microspheres, Spheres, Suspension of Particles, Collisions, Sedimentation, Stirring of Components, Shear Forces, Isothermal Processing, Nucleation, Particle Growth, Particle Size Distribution, Buoyancy Effects Diminished, Bubbles, Bubble Formation, Contained Fluids,

Viscosity, Piston System, Volume Change, Volume Compensation, Air Fan, Vacuum, Hardware Malfunction, Incomplete Sample Processing, Processing Difficulties, Acceleration Effects, Rocket Vibrations

Number of Samples: four
Sample Materials: Monomer: styrene, water; Latex #9: seed diameter = 5.6 microns at 2/1 monomer/polymer ratio; Latex #10 seed diameter = 5.6 microns at 4/1 monomer/polymer ratio; Latex #11: seed diameter = 5.6 microns at 6/1 monomer/polymer ratio; Latex #12: seed diameter: 0.19 microns at 2/1 monomer/polymer ratio; buffer (Latex 12 only): sodium bicarbonate; initiator (Latex 12 only): potassium persulfate
Container Materials: stainless steel

Experiment/Material Applications:
See Vanderhoff, STS-003.

References/Applicable Publications:
In addition to the publications listed below, please refer to Vanderhoff, STS-003 and STS-004 for listings of additional publications.

(1) Input received from Co-Investigator D. Kornfeld, September 1989 and July 1993.

(2) Kornfeld, D. M.: Monodisperse Latex Reactor (MLR) - A Materials Processing Space Shuttle Middeck Payload. NASA TM-86487, January 1985. (post-flight)

(3) STS-6 Sixth Space Shuttle Mission, April 1983, NASA Press Kit, pp. 4 and 38-39. (preflight)

(4) STS-7 Seventh Space Shuttle Mission, June 1983, NASA Press Kit, p. 51. (briefly discusses STS-6 successful results)

(5) "First Space Product Set to be Developed for Commercial Use." NASA Activities, August 1984, Vol. 15, No. 8, pp. 5-6. (post-flight)

(6) Monodisperse Latex Reactor (MLR). Application Payload Projects, Spacelab Payload Project Office, Marshall Space Flight Center, Huntsville, Alabama, 8 pp. (document developed by Teledyne Brown Engineering)

(7) Sudol, E. D., El-Aasser, M. S., and Vanderhoff, J. W.: Kinetics of Successive Seeding of Monodisperse Polystyrene Latexes I. Initiation via Potassium Persulfate. Accepted for publication in J. Polym. Sci., 1986. <Note: The current status of this document is unclear at this time.>

(8) Sudol, E. D., El-Aasser, M. S., and Vanderhoff, J. W.: Kinetics of Successive Seeding of Monodisperse Polystyrene Latexes II. Azo Initiators with and without Inhibitors. Accepted for publication in J. Polym. Sci., 1986. <Note: The current status of this publication is unclear at this time.>

(9) Vanderhoff, J. W., El-Aasser, M. S., Micale, F. J., Sudol, E. D., Tseng, C.-M., Sheu, H.-R., and Kornfeld, D. M.: The First Products Made in Space: Monodisperse Latex Particles. AIAA 25th Aerospace Sciences Meeting, January 12-15, 1987, Reno, Nevada, AIAA Paper #87-0389.

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Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Middeck Experiment, Crew Compartment
Processing Facility: Monodisperse Latex Reactor (MLR) (same apparatus as employed on the earlier STS-006 flight but now with minor electronics changes to permit slower stirring speeds)
Builder of Processing Facility: Reactors: General Electric Company, Valley Forge, Pennsylvania; Support Electronics Package (SEP): Rockwell International, Downey, California

Experiment:
Monodisperse Latex Reactor (MLR-4)

This STS-007 experiment was the fourth in a series of investigations designed by Vanderhoff et al. to study the low-gravity production of monodisperse polystyrene latex microspheres (see Vanderhoff, STS-003, STS-004, STS-006).

The Monodisperse Latex Reactor (MLR), which had been previously employed to produce latex particles on STS-003, STS-004 and STS-006 was reused during this STS-007 mission. The STS-007 latex production experimental procedure was essentially the same as that of the earlier STS-003 mission (see Vanderhoff, STS-003).

During this STS-007 experiment, four space-based polymerization experiments were conducted using different seed sizes and monomer-polymer ratios: latex number 13 used 7.9 micron seeds at a monomer/polymer ratio of 6:1; latex number 14 used 10.3 micron seeds at a monomer/polymer ratio of 4:1; latex number 15 used 10.3 micron seeds at a monomer/polymer ratio of 6:1; latex number 16 used 10.3 micron seeds at a monomer/polymer ratio of 6:1. Latexes 13 and 14 had the same pre-processing and processing stirring speeds (both speeds 13 rpm); latex 15 had a stirring speed of 13 rpm (pre-processing) and 6 rpm (processing); latex 16 had a stirring speed of 6 rpm (pre-processing) and 3 rpm (processing). The seed latex used for latex 13 (7.9 microns in diameter) had been produced in space during the STS-006 experiment (see Vanderhoff, STS-006).

Post-flight examination of the latex particles indicated that all four flight experiments resulted in an excellent latex product. The coefficients of size variation (defined as the standard deviation divided by the number-average particle diameter) for the flight materials were slightly smaller than for the seed particles and significantly smaller than those produced on Earth. All flight latexes contained a small number of offsize (smaller and larger) particles.

The conversion-time curves for flight latexes 13 and 16 were practically the same as the corresponding ground-based materials. The curves for flight latexes 14 and 15 were slightly above those of the ground-based, control samples. "The leveling-off of the conversion-time curves was attributed to the formation of a nitrogen bubble or sticking of the dilatometer." (9, p. 6) This characteristic was also observed for the latexes produced during the STS-006 experiment. It was also reported that temperature gradients existed between the cylinder wall and center of the dilatometer. These gradients increased with increasing particle size and monomer-polymer ratio.

Key Words: Technological Experiments, Emulsion Polymerization, Monodisperse Latex Particles, Polystyrene Latex Microspheres, Spheres, Collisions, Suspension of Particles, Stirring of Components, Shear Forces, Nucleation, Particle Growth, Particle Size Distribution, Sedimentation, Thermal Gradient, Buoyancy Effects Diminished, Bubbles, Bubble Formation, Contained Fluids, Viscosity, Piston System, Volume Compensation, Volume Change, Vacuum

Number of Samples: four
Sample Materials: Monomer: styrene, water; Latex #13: seed diameter = 7.9 microns at 6/1 monomer/polymer ratio; Latex #14: seed diameter = 10.3 microns at 4/1 monomer/polymer ratio; Latex #15: seed diameter = 10.3 microns at 6/1 monomer/polymer ratio; Latex #16: seed diameter: 10.3 microns at 6/1 monomer/polymer ratio
Container Materials: stainless steel

Experiment/Material Applications:
See Vanderhoff, STS-003.

References/Applicable Publications:

(1) Input received from Co-Investigator D. Kornfeld, September 1989 and July 1993.

(2) Kornfeld, D. M.: Monodisperse Latex Reactor (MLR) - A Materials Processing Space Shuttle Middeck Payload. NASA TM-86487, January 1985. (post-flight)

(3) STS-7 Seventh Space Shuttle Mission, June 1983, NASA Press Kit, pp. 51-52.

(4) "First Space Product Set to be Developed for Commercial Use." NASA Activities, August 1984, Vol. 15, No. 8, pp. 5-6. (post-flight)

(5) Monodisperse Latex Reactor (MLR). Application Payload Projects, Spacelab Payload Project Office, Marshall Space Flight Center, Huntsville, Alabama, 8 pp. (document developed by Teledyne Brown Engineering)

(6) Vanderhoff, J. W., El-Aasser, M. S., Micale, F. J., Sudol, E. D., Tseng, C.-M., Silwanowicz, A., and Kornfeld, D. M.: Preparation of Large-Particle-Size Monodisperse Latexes in Space. J. Dispersion Sci. technology, Vol. 5, 1984, pp. 231-246. (post-flight)

(7) Sudol, E. D., El-Aasser, M. S., and Vanderhoff, J. W.: Kinetics of Successive Seeding of Monodisperse Polystyrene Latexes I. Initiation via Potassium Persulfate. Accepted for publication in J. Polym. Sci., 1986. <Note: The current status of this document is unclear at this time.>

(8) Sudol, E. D., El-Aasser, M. S., and Vanderhoff, J. W.: Kinetics of Successive Seeding of Monodisperse Polystyrene Latexes II. Azo Initiators with and without Inhibitors. Accepted for publication in J. Polym. Sci., 1986. <Note: The current status of this document is unclear at this time.>

(9) Vanderhoff, J. W., El-Aasser, M. S., Micale, F. J., Sudol, E. D., Tseng, C.-M., Sheu, H.-R., and Kornfeld, D. M.: The First Products Made in Space: Monodisperse Latex Particles. AIAA 25th Aerospace Sciences Meeting, January 12-15, 1987, Reno, Nevada, AIAA Paper #87-0389.

See Vanderhoff, STS-003 and STS-004 for listings of additional publications.

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Experiment Origin: USA
Mission: STS Launch #10, STS-011 (STS 41-B Challenger)
Launch Date/Expt Date: February 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Middeck Experiment, Crew Compartment
Processing Facility: Monodisperse Latex Reactor (MLR) (same apparatus as used on STS-007)
Builder of Processing Facility: Reactors: General Electric Company, Valley Forge, Pennsylvania; Support Electronics Package (SEP): Rockwell International, Downey, California

Experiment:
Monodisperse Latex Reactor (MLR-5)

This STS-011 experiment was the fifth in a series of investigations designed by Vanderhoff et al. to study the low-gravity production of monodisperse polystyrene latex microspheres (see Vanderhoff, STS-003, STS-004, STS-006, STS-007).

The Monodisperse Latex Reactor (MLR), which had been employed during the previous experiments initiated by Vanderhoff, was reused during the STS-011 mission. The STS-011 latex production experimental procedure was essentially the same as that of the earlier STS-003 mission (see Vanderhoff, STS-003).

During this STS-011 experiment, four polymerization experiments were conducted using different seed sizes and monomer-polymer ratios: latex number 17 contained seeds of 17.8 microns and had a monomer/polymer ratio of 5:1; latex number 18 contained seeds of 17.8 microns and had a monomer-polymer ratio of 5:1; latex number 19 contained seeds of 10.3 microns and had a monomer-polymer ratio of 6:1; latex number 20 contained seeds of 10.3 microns and had a monomer-polymer ratio of 6:1. The seed latexes used for flight latexes 17 and 18 (17.8 microns in diameter) had been produced in space during the STS-007 experiment (see Vanderhoff, STS-007). Latexes 17 and 19 had pre-processing and processing stirring speeds of 13 rpm and 6 rpm, respectively. Latexes 18 and 20 had pre-processing and processing stirring speeds of 6 rpm and 3 rpm, respectively.

Reportedly, after the shuttle landed and "For several hours before unloading, the MLR was inverted periodically to redisperse the settled latex particles. When the stirrers were turned on, the movement of the flight latex 19 stirrer was restricted; therefore, it was turned off immediately; the dilatometer had a broken stirrer-shaft shear-pin and it contained a mass of coagulum between one side of the stirrer blade and the wall. It is not known whether the formation of coagulum stalled the stirrer and broke the shear pin or the failure of the shear pin caused the formation of coagulum; flight latex 20, which was identical except for the stirring rates, contained no coagulum, yet failure analysis of the broken shear pin [from flight latex 19] showed no evidence of fatigue failure." (9, p. 6) It was also reported that no ground-based control experiments were carried out because earlier experiments (STS-006 and STS-007) indicated that "[on the ground]...the coagulum increased with increasing particle size so strongly that the valuable seed latex would have been wasted." (9, p. 6)

All of the flight latexes contained offsize particles (smaller and larger). "The smaller offsize particles were removed by repeated sedimentation-decantation. The numbers of offsize larger particles determined by optical microscopy were about twice those of the seed latexes." (9, p.9) <Note: The intent was to grow each existing large seed ball to a larger size without nucleating a new crop of submicron-size (offsize) balls. In this case, a new crop of such balls formed (see Vanderhoff, STS-003, for additional information on such particle nucleation).>

The product particle sizes were (1) 30.4 microns for latex 17, (2) 30.9 microns for latex 18, (3) 18 microns for latex 19, and (4) 19.4 microns for latex 20. "Flight latexes 17 and 18 (30 microns) were accepted by the National Bureau of Standards as a Standard Reference Material, the second product made in space for sale on earth. These particles were also found to be more perfect spheres than the ground-based particles." (9, p. 6)

The conversion-time curves for all the flight latexes virtually coincided. Data from latexes 17 and 18 showed a slightly greater deviation (upward) from linearity than latexes 19 and 20, "...which was attributed to the higher monomer-polymer ratio [latexes 19 and 20] and hence lower viscosity delaying the onset of autoacceleration." (9, p. 6)

Results from all of the low-gravity MLR experiments (latexes 1-20) confirmed the original rationale of the experiments:

(1) Flight polymerizations resulted in negligible amounts of coagulum compared to the significant amounts in ground-based experiments.

(2) The latexes produced in space had a narrower particle size distribution than those produced on Earth.

(3) The particles from the low-gravity experiments were more spherical than those produced on Earth.

(4) The number of offsize larger particles was greater for the ground-based latexes than for the flight latexes.

(5) Completion of the polymerization of the partially polymerized STS-004 low-gravity latexes resulted in a broadened particle size distribution and more offsize larger particles.

(6) The polymerization rates, within experimental error, were the same in space as on Earth.

Key Words: Technological Experiments, Emulsion Polymerization, Monodisperse Latex Particles, Polystyrene Latex Microspheres, Spheres, Sphericity, Suspension of Particles, Collisions, Stirring of Components, Shear Forces, Sedimentation, Isothermal Processing, Nucleation, Particle Growth, Particle Size Distribution, Coagulation, Buoyancy Effects Diminished, Viscosity, Contained Fluids, Piston System, Volume Change, Volume Compensation, Vacuum, Hardware Malfunction

Number of Samples: four
Sample Materials: Monomer: styrene, water; Latex #17: seed diameter = 17.8 microns at 5/1 monomer/polymer ratio; Latex #18: seed diameter = 17.8 at 5/1 monomer/polymer ratio; Latex #19: seed diameter = 10.3 microns at 6/1 monomer/polymer ratio; Latex #20: seed diameter 10.3 microns at 6/1 monomer/polymer ratio
Container Materials: stainless steel

Experiment/Material Applications:
See Vanderhoff, STS-003.

References/Applicable Publications:
(1) Input received from Co-Investigator D. Kornfeld, September 1989 and July 1993.

(2) Kornfeld, D. M.: Monodisperse Latex Reactor (MLR) - A Materials Processing Space Shuttle Middeck Payload. NASA TM-86487, January, 1985. (post-flight)

(3) 41-B Tenth Space Shuttle Mission, February 1984, NASA Press Kit, p. 30. (very brief summary)

(4) "First Space Product Set to be Developed for Commercial Use." NASA Activities, August 1984, Vol. 15, No. 8, pp. 5-6. (post-flight)

(5) Monodisperse Latex Reactor (MLR). Application Payload Projects, Spacelab Payload Project Office, NASA Marshall Space Flight Center, Huntsville, Alabama, 8 pp. (document prepared by Teledyne Brown Engineering; post-flight)

(6) Vanderhoff, J. W., El-Aasser, M. S., Micale, F. J., Sudol, E. D., Tseng, C.-M., Silwanowicz, A., and Kornfeld, D. M.: Preparation of Large-Particle-Size Monodisperse Latexes in Space. J. Dispersion Sci. technology, Vol. 5, 1984, pp. 231-246. (post-flight)

(7) Sudol, E. D., El-Aasser, M. S., and Vanderhoff, J. W.: Kinetics of Successive Seeding of Monodisperse Polystyrene Latexes I. Initiation via Potassium Persulfate. Accepted for publication in J. Polym. Sci., 1986. <Note: The current status of this document is unclear at this time.>

(8) Sudol, E. D., El-Aasser, M. S., and Vanderhoff, J. W.: Kinetics of Successive Seeding of Monodisperse Polystyrene Latexes II. Azo Initiators with and without Inhibitors. Accepted for publication in J. Polym. Sci., 1986. <Note: The current status of this document is unclear at this time.>

(9) Vanderhoff, J. W., El-Aasser, M. S., Micale, F. J., Sudol, E. D., Tseng, C.-M., Sheu, H.-R., and Kornfeld, D. M.: The First Products Made in Space: Monodisperse Latex Particles. AIAA 25th Aerospace Sciences Meeting, January 12-15, 1987, Reno, Nevada, AIAA Paper #87-0389.

See Vanderhoff, STS-003 and STS-004 for additional publications.

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Experiment Origin: USA
Mission: STS Launch #6, STS-006 (STS 31-B, Challenger)
Launch Date/Expt. Date: April 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: College Student Experiment; NASA Get Away Special (GAS) canister G-049
  Volume of Canister 5.0 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-049: The United States Air Force Academy, Colorado Springs, Colorado
Processing Facility: Plexiglas™ box configured with copper anode and copper cathode.
Builder of Processing Facility: Unknown

Experiment:
Electroplating

On Earth, gravitational forces dictate the distribution of metal during electroplating. In space, a more even plating of metal may be possible.

This STS experiment was one of six investigations housed within the G-049 Get Away Special canister on STS-006. Four other experiments (of the six) were applicable to this data base (see Amidon, STS-006 (Chapter 14); Gross, STS-006 (Chapter 18); Neel, STS-006 (Chapter 4); Streb, STS-006 (Chapter 14)). The specific objective of the electroplating experiment was to evenly plate one metal (ionically) on to another metal.

<Note: Brief descriptions of the experimental setup were provided in Reference (1) (a document published prior to the launch of STS-006) and Reference (2) (a document published after the return of the shuttle). Because Reference (2) did not verify all of the vital information provided in Reference (1), the actual experimental setup is unclear. However, it appears that prior to launch, a copper rod (anode), suspended within a copper cylinder (cathode) was placed inside a Plexiglas™ box filled with an electrolyte solution (0.5 molar copper sulfate).>

During the mission, an electrical current was applied across the solution.

<Note: Although Reference (2) indicated that sometime during the mission the solution would freeze (and provisions had been made to handle the accompanying volume increase), the reference did not specifically state why the solution would freeze. In addition, this reference indicated that a heater was used during the experiment, but did not detail why the apparatus was used. Thus, it was presumed that (1) the solution would freeze in the cold payload bay and (2) the solution would have to be warmed by the heater.>

The experimental results, as reported were somewhat unclear: "In orbit, the solution did freeze (the heater did not work as expected) and electroplating did result from a current across the solution; however, the magnitude of the electroplating was of the level of two hours, not the hoped-for two days. The major conclusion was that electroplating can occur in [a] vacuum or gravity-free environment."

No further information could be located which described this experiment.

Key Words: Technological Experiments, Electroplating, Plating, Metals, Anode, Cathode, Electrodes, Electrolyte Solution, Coated Surfaces, Electric Field, Contained Fluids, Volume Compensation, Volume Change, Freezing, Vacuum, Processing Time Not As Long As Planned

Number of Samples: one
Sample Materials: copper anode and cathode; electrolyte solution: 0.5 molar copper sulfate
(Cu*S*)
Container Materials: Plexiglas™ box

Experiment/Material Applications:
Direct applications of this research were not cited in the available publications. However, it is suspected that the electroplating was studied to determine if more homogeneous surface coatings (with improved characteristics) could be created in the low-gravity environment.

References/Applicable Publications:

(1) Cargo Systems Manual: GAS Annex for STS-6, JSC-17645 Annex STS-6, December 3, 1982. (very short description; preflight)

(2) Swan, P. and Worsowicz, C.: The Eaglets have Flown. Space Education, Vol. 1, No. 7, May 1984, pp. 317-319. (post-flight)

(3) STS-6 Getaway Specials, NASA News, NASA GSFC, November 24, 1982. (preflight)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report # EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(5) NASA STS-6 Sixth Space Shuttle Mission Press Kit, April 1983, pp. 41-43. (preflight)

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Experiment Origin: USA
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Spacelab Rack, Rack Number #7
Processing Facility: Fluids Wetting and Spreading (FWS) Module
Builder of Processing Facility: Unknown

Experiment:
Tribological Experiments in Zero Gravity (INT011)

The wetting and spreading of a liquid over a solid surface is governed by both gravity-dependent and gravity-independent forces. In space, the gravity-dependent forces are greatly reduced, thus facilitating the study of wetting phenomenon controlled by interfacial and capillary forces.

This Spacelab 1 experiment was designed to study the wetting, spreading and operational behavior of bearing lubricants under low-gravity conditions. The experiment consisted of two major investigations: (1) The Fluid Wetting and Spreading Study (FWS) and (2) The Journal Bearing Study. Details of these two investigations are discussed separately within this experiment summary. <Note: It appears that all of the equipment required for these two investigations was contained within a drawer in Spacelab Rack #7 (including a camera, accelerometer, power supply, wetting surfaces, rotor, etc.). See Reference (4) for a schematic of the drawer layout.>

<Note: At the time this experiment summary was prepared, Reference (1) was the only document detailing postflight results which could be obtained. These results are reproduced below almost in their entirety because the information as written was difficult to follow and no accompanying photographs of the surfaces/fluid-wetting accompanied the results.>

The Fluid and Wetting Study

The useful life of a ball bearing is often dependent on the wetting and spreading behavior of the bearing lubricant on the bearing. The fluid wetting and spreading study (FWS) addressed the behavior of a lubricant drop on various surfaces.

The description of the experimental apparatus, employed fluids, and employed surfaces was reported as follows: "The FWS module is a mechanized fluid-dispensing device. A separate unit is used for each of four selected test fluids. Each module contains three geometrically identical surface specimens. Twelve fluid-surface combinations were used during the Spacelab 1 flight." (1, p. 202)
"The study was conducted by photographing the wetting and spreading process as soon as the test fluid "surfaces" on the specimen. In each test sequence, approximately 24 μl of the test fluid was displaced through the central hole of each test specimen. Filming started at 24 frames per second for 8 seconds, then changed to one frame per second for 8 minutes. All solid specimens were made of 440C stainless steel with various finishing and treatment conditions.... An oblique mirror beside each specimen showed the side profile." (1, p. 202)

Spreading and wetting results of only one of the test fluids were presented in the available references: "Cinematographic records of wetting and spreading of SRG-10 [paraffinic] oil were analyzed by measuring the mean radius of the wetted spot at various times. Three phenomenological regimes were revealed by the spreading history:

"1) Radius of each wetted spot grew to 0.15 cm in about 0.08 seconds with same rate on all three specimens.

"2) The wet radius on the barrier film-coated specimen [fluoropolymer in solution with prefluorinated cyclic ether (MIL-B-81744)] grew to 0.2 cm at the end of 0.8 second. The side profile was roughly hemispheric. Wet radii on the other two specimens increased to 0.3 cm in the same period.

"3) The wet radius on the barrier film-coated specimen stopped changing. Spreading continued on the other two specimens, with the wet radii reaching 0.50 and 0.38 cm, respectively, on the prewetted and clean specimens in 440 seconds. The spot shape on the prewetted specimen became oblong. A milli-g acceleration disturbance in this period has been established.

"These results suggest that SRG-10 tends to wet and spread on a clean solid surface similar to 440C stainless steel, that the rate of the advance of the contact line on a clean surface is

very slow, and that barrier film coating can effectively prevent migration of the oil." (1, p. 202)

It was reported that analysis of other photographic records was still underway.

The Journal Bearing Study

"In the normal functioning of an oil-lubricated journal bearing, the clearance space is only partially filled. On the unleaded side of the journal, natural drainage leaves behind a two-phase film which is connected to an outboard void space. Stable operation of a liquid-lubricated journal bearing is known to be dependent on the presence of the two-phase film." (1, p. 202)

This Spacelab journal bearing study investigated the morphology of the two-phase film subjected to shearing forces between to surfaces.

"The journal bearing module has a symmetrical, rigid rotor supported by a pair of experimental journal bearings. Three bearing configurations were used to control various kinematic and geometric parameters. In the first configuration, the bearings were of plain cylindrical geometry, and the rotor could be fitted with an unbalanced mass to yield an acceleration of 0.77 g. In the second, the bearings were shaped by three centrally preloaded arcs. In the third, the bearings were similar to those in the first configuration, but one end of the shaft was also fitted with a ball bearing to fix the operating eccentricity to three-fourths of the radial clearance at that end.

"The experimental bearings are made of glass to permit viewing of the two-phase film. Inclined mirrors on either side of each bearing provide a full view. Encoding markers on the rotor are sensed optically to monitor speed and to furnish triggering signals for the camera and synchronized stroboscopic lighting.

"The journal bearing module operates in the coast-down mode. The drive mechanism is disengaged when the rotor speed reaches 600 rev/min. The camera drive is synchronized with the rotor so that events related to unavoidable variations around the journal surface remain unchanged in successive cinematographic views. Non-contact proximity sensors along two mutually perpendicular axes are used to monitor the radial motion of the rotor.

"The cinematographic records showed the following results, which correspond to the three bearing configurations described above:

"1) With the balanced rotor, a streamer structure which almost wraps around the full circumference is seen. The appearance fluctuates and repeats in approximately every other frame. This suggests the presence of a one-half rotational rate oscillation. The possibility of such a flow structure was previously postulated.... This general appearance continues as the rotor slows down to about 200 rev/min. The void content of the streamer structure is somewhat reduced at the lower speeds. With the unbalanced rotor, the two-phase morphology changes with speed. At the higher speeds, there is a more pronounced circumferential variation in the flow structure in each frame, but the frame-to-frame fluctuation is less apparent. When the rotor speed falls below about 300 rev/min, the general appearance reverts back to that seen for the balanced rotor.
"2) Well-defined void regions are fixed to the three-arc geometry. Occasionally, small isolated voids are seen going around. There are no significant frame-to-frame fluctuations.

"3) The general appearance is similar to that in the second case, except that fixed void regions are less extensive and are not divided into three groups.

Data from the proximity sensors remain to be analyzed." (1, p. 202)

<Note: No other details concerning two experiments could be located at this time. (Reference (3) could not be obtained at the time this experiment summary was written.)>

Key Words: Technological Experiments, Tribology, Fluid Physics, Bearings, Lubricants, Wetting, Wetting Kinetics, Surface Tension, Free Surface, Meniscus Shape, Interfacial Tension, Liquid Spreading, Interface Physics, Capillary Forces, Capillary Flow, Solid/Liquid Interface, Drops, Coated Surfaces, Liquid Transfer, Liquid Expulsion Through a Small Orifice, Shear Forces, Sample Rotation, Thin Films, Two-Phase System, Multiphase Media, Gaps, Acceleration Effects, Acceleration Measurements

Number of Samples: not applicable
Sample Materials: Liquid Lubricants: SRG-10 Superrefined Paraffinic Oil; Bray 815Z, vacuum-distilled perfluoroalkyl polyether; Apiezon Cn, molecularly distilled paraffinic; 90:10 blend of four and five-ring polyphenyl ethers.
Surface Material: 440C stainless steel, three finishes (ground or polished): clean, prewetted, and barrier film coated. Bearing Material: Glass (see Reference (1) for details)

Container Materials: not applicable

Experiment/Material Applications:
The specific reason why each fluid/surface combination was chosen was not presented in the available references. However, it is believed that the materials are typical for bearing applications.

References/Applicable Publications:
(1) Pan, C.H.T., Gause, R. L., and Whitaker, A. F.: Tribology Experiment in Zero Gravity. Science, Vol. 225, July 13, 1984, pp. 202-203. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements)

(3) Todd, M. J.: Activities Report in Space Tribology Progress Report 1984, Paris, 1985 16 pp, ESA CR(P)-2043. (post-flight)

(4) Pan, C.H.T., Whitaker, A. F., and Gause, R. L.: Wetting, Spreading and Operating Characteristics of Bearing Lubricants in a Zero Gravity Environment. In Spacelab Mission 1 Experiment Descriptions - 2nd Edition, NASA TM-82448, November 1981, pp. III-17 - III-20. (preflight)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #10, STS-011 (STS 41-B, Challenger)
Launch Date/Expt Date: February 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: West German Get Away Special (GAS) MAUS Canister DG-200A
Volume of Canister: 5.0 cubic feet
Location of Canister: The West German Shuttle Pallet Satellite (SPAS-01A) (SPAS was a small experiment carrier removed from the STS payload bay by the Canadian Remote Manipulator Arm, but remained attached to the arm throughout the mission at a position overhead about 5 to 10 feet from the forward payload bay bulkhead. The satellite was returned to the cargo bay before the shuttle's return.)
Primary Developer/Sponsor of DG-200A: The German Ministry of Research and Technology (BMFT)/Messerschmitt-Boelkow-Blohm (MBB-ERNO)
Processing Facility: Slip Casting Thermostat Furnace (aluminum block into which 13 holes had been drilled to accommodate 13 samples)
Builder of Processing Facility: Motoren und Turbinen Union (MTU), Munich, Germany

Experiment:
<u>Slip Casting I</u>

During slip casting, a ceramic slurry is used to form complicated shapes of hollow bodies. On Earth, the effects of gravity-induced sedimentation and hydrostatic pressure limit the materials which can be employed for the process. Specifically, materials must have (1) constituents of equal densities or (2) stabilizing additives. A stabilizing additive can have detrimental effects on the final properties of the cast material.

This Space Shuttle STS-011 experiment was the first in a series of investigations designed by Schweitzer to study slip casting under low-gravity conditions. The specific objective of the experiment was to demonstrate (with model materials) that slip casting of unstabilized slurries can be accomplished in a low-gravity environment.

Prior to the shuttle mission, 13 flight samples were prepared. <Note: It appears that the compositions of these samples were the same as those employed on during Schweitzer's later experiment

(see Schweitzer, STS-025). If so, the following description of the samples would be correct:> Each sample consisted of a mixture of ceramic and/or metal powders kneaded into paraffin wax. The majority of the samples had either (1) only similar diameter, micron-sized Al powders (20, 40, or 60 vol.%); (2) a mixture of two different micron-sized Al particles (two different vol.%); (3) only similar micron-sized Al_2O_3 powders (40 vol.%); (4) a mixture of similar diameter, micron-sized Al and Al_2O_3 powders (similar vol.% of each); (5) mixtures of Al_2O_3 powders and (a) W powders or (b) Mo powders (similar and different vol.%, similar and different micron sizes). <Note: In addition to the powders listed above, Reference (9) also mentions "3 mm Schlicker 1, 3 mm Schlicker 7, Schlicker 6, APK7, and Schlicker 12." These powders are unfamiliar to the editors.> (Specific compositions of each sample can be found in Reference (9).) Rods of these solid slurries were pressed into cartridges against the ends of porous ceramic disks. (The disks were mounted in the lower halves of the cartridges.) The cartridges were configured in the Slip Casting Thermostat furnace contained within the MAUS DG-200 Get Away Special Canister (see References (4) and (6) for details of the MAUS program).

During the STS-011 experiment, the Slip Casting Thermostat furnace was to be used to process several samples. Details of the expected melting and solidification procedure of the samples were similar to those procedures realized during Schweitzer's later experiment on STS-025.

It was reported that when a relay controlling the battery power to the furnace failed to open and the controller of the safety circuits sensed this problem, an emergency shutdown of the experiment was invoked. Thus, the MAUS payload could not be activated and no results from the experiment were attained. The experiment was reflown on STS-025 (see Schweitzer, STS-025).

<Note: References (3) and (9) were not translated prior to the preparation of this experiment summary; therefore, only small portions of the information contained therein could be evaluated.>

Key Words: Technological Experiments, Slip Casting, Casting, Melt and Solidification, Model Materials, Slurry Solutions, Suspension, Density Difference, Particle Dispersion, Sedimentation, Hydrostatic Pressure, Powder Metallurgy, Ceramics, Porous Material, Hardware Malfunction, Sample Not Processed As Planned

Number of Samples: thirteen
Sample Materials: Ceramic and metal powders with different grain size kneaded into solid paraffin wax. See above Experiment section for more details.
(Al*, Al*O*, W*, Mo*)
Container Materials: aluminum
(Al*)

Experiment/Material Applications:
Ceramic and metal powders in wax are models of unstabilized slurries.

The specific uses of other (non-model) slip cast materials were not discussed in the available publications.

References/Applicable Publications:
(1) Otto, G. H. and Baum, D.: Material Sciences Experiments Under Microgravity Conditions with M*A*U*S. In 1985 Get Away Special Experimenter's Symposium, October 8-9, 1985, Goddard Space Flight Center, Greenbelt, Maryland, NASA CP-2401, pp. 101-108. (post-flight)

(2) STS-11 Cargo Systems Manual: SPAS 01A, JSC-19272 Basic Version, NASA JSC, September 15, 1983. (preflight)

(3) Schweitzer, K., Lackermeier, R., and Track, W.: Schlickergiessen unter Schwerelosigkeit. Abschlussbericht, Reference No. 01QV320-ZK-SN-SLN 8802 (1987). (in German, English abstract)

(4) Otto, G. H.: Experimental Results from Automated MAUS Payloads. IAF Paper #88-351 (1988).

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(7) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(8) Input received from Principal Investigator K. Schweitzer, August 1993.

(9) Track, W., Schweitzer, K. K., and Lackmeier, R.: Schlickergiessen unter den Bedingungen der Schwerelosigkeit im Weltraum. In Proceedings: H. Kolaska, H. Grewe, "Moderne Formgebungserfahren, Pulvermetallurgie-Keramik" (Symposium, Hagen, November 14-15, 1985), pp. 73-92, Meisenheim, 1985, ISBN 3-925543-00-7. (post-flight, in German)

(10) Feazel, M.: Shuttle Will Fully Deploy German Pallet. Aviation Week & Space Technology, February 21, 1983, pp. 67-68.

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #18, STS-025 (STS 51-G, Discovery)
Launch Date/Expt Date: June 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: West German Get Away Special (GAS) MAUS Canister DG-200B (also designated as NASA Get Away Special (GAS) Canister G-027)
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of DG-200B/G-027: The German Ministry of Research and Technology (BMFT)/Messerschmitt-Boelkow-Blohm (MBB-ERNO)
Processing Facility: Slip Casting Thermostat Furnace (aluminum block into which 13 holes had been drilled to accommodate 13 samples)
Builder of Processing Facility: Motoren und Turbinen Union (MTU), Munich, Germany

Experiment:
Slip Casting II

This STS-025 experiment was the second in a series of investigations designed by Schweitzer to study slip casting under low-gravity conditions (see Schweitzer, STS-011). The specific objective of the experiment was to demonstrate (with model materials) that slip casting of unstabilized slurries can be accomplished in a low-gravity environment.

Prior to the shuttle mission, 13 flight samples were prepared. The samples consisted of a mixture of ceramic and/or metallic powders kneaded into paraffin wax. The majority of the samples had either (1) only similar diameter, micron-sized Al powders (20, 40, or 60 vol.%); (2) a mixture of two different micron sized Al particles (two different vol.%); (3) only similar micron-sized Al_2O_3 powders (40 vol.%); (4) a mixture of similar diameter, micron-sized Al and Al_2O_3 powders (similar vol.% of each); (5) mixtures of Al_2O_3 powders and (a) W powders or (b) Mo powders (similar and different vol.%, similar and different micron sizes). <Note: In addition to the powders listed above, Reference (11) also mentions "3 mm Schlicker 1, 3 mm Schlicker 7, Schlicker 6, APK7, and Schlicker 12." These powders are unfamiliar to the editors.> (Specific compositions of each sample can be found in Reference (11).) Rods of these solid slurries were pressed into cartridges against the ends of porous ceramic

suction disks. (The disks were mounted in the lower halves of the cartridges.) The cartridges were configured in the Slip Casting Thermostat furnace contained within the MAUS DG-200B Get Away Special canister (see References (3) and (7) for details of the MAUS program).

At the initiation of the STS-025 experiment, the upper sections of the cartridges were heated, melting the solid slurries. "Then the slip casting process was started by additionally heating the lower part of the cartridge containing suction bodies made of porous ceramic. These [bodies] did slowly [partially] absorb paraffin [via capillary forces of the porous ceramic] but not the dispersed particles. The casting process was stopped by turning off the furnace and cooling the samples. Solidification of the paraffin did preserve the slip cast layers as well as the residual slurries for later examination on Earth in respect to their structure and particle distribution." (7, p. 5)

It was reported that the experiment was not performed as anticipated. "Due to a temperature excess of the upper heater the programmed temperature profile could not be executed properly during the mission. To... [analyze] the impact of this malfunction on the samples the complete furnace including [the] specimens has been examined by means of computer tomography. Undesired gas bubbles were formed and their presence disturbed the solidification front. The slip castings could therefore not be characterized in the desired manner. However, it could be shown that particles are transported to form slip casting layers and that this process can be performed under weightlessness with unstabilized suspensions." (7, p. 5)

It was further reported: "Small particles are transported more easily and are filling the interstices between the larger grains. At particle concentrations > 60 vol.-%, no further densification is observed." (6, abstract)

<Note: References (6) and (11) were not translated prior to the preparation of this experiment summary; therefore, only small portions of the information contained therein could be evaluated. No other information (published in English) concerning this experiment could be located at this time.>

Key Words: Technological Experiments, Slip Casting, Melt and Solidification, Model Materials, Slurry Solutions, Suspension, Sedimentation, Powder Metallurgy, Ceramics, Particle Dispersion, Particle Distribution, Particle Transport, Grain Size, Bubbles, Bubble Formation, Solidification Front Physics, Porous Material,

Capillary Forces, Processing Difficulties, Thermal Environment More Extreme Than Predicted

Number of Samples: thirteen
Sample Materials: Ceramic and metal powders with different grain size kneaded into solid paraffin wax. See above Experiment section for more details.
(Al*, Al*O*, W*, Mo*)
Container Materials: aluminum
(Al*)

Experiment/Material Applications:
See Schweitzer, STS-011.

References/Applicable Publications:

(1) Cargo Systems Manual: GAS Annex for STS 51-G, JSC-17645 51-G, Rev-A, March 20, 1985. (short description; preflight)

(2) Space Shuttle Mission 51-G Press Kit, June 1985. (preflight)

(3) Otto, G. H. and Baum, D.: Material Sciences Experiments Under Microgravity Conditions with MAUS. In Goddard Space Center's 1985 Get Away Special Experimenter's Symposium, October 8-9, 1985, pp. 101-108, NASA CP-2401. (preflight)

(4) Otto, G. H. and Staniek, S.: Recent Results from MAUS Payloads. In NASA Goddard Space Flight Center's 1986 Get Away Special Experimenter's Symposium, October 7-8, 1986, NASA CP-2438. (post-flight)

(5) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, the NASA GAS Team, 1989, p. 29. (post-flight; very brief description)

(6) Schweitzer, K., Lackermeier, R., and Track, W.: Schlickergiessen unter Schwerelosigkeit. Abschlussbericht, Reference No. 01QV320-ZK-SN-SLN 8002 (1987). (in German, English abstract)

(7) Otto, G. H.: Experimental Results from Automated MAUS Payloads. IAF Paper #88-351 (1988).

(8) Input received from MAUS Project Scientist G. Otto, October 1989 and August 1993.

(9) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report # EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(10) Input received from Principal Investigator K. Schweitzer, August 1993.

(11) Track, W., Schweitzer, K. K., and Lackmeier, R.: Schlickergiessen unter den Bedingungen der Schwerelosigkeit im Weltraum. In Proceedings: H. Kolaska, H. Grewe, "Moderne Formgebungserfahren, Pulvermetallurgie- Keramik" (Symposium, Hagen, November 14-15, 1985), pp. 73-92, Meisenheim, 1985, ISBN 3-925543-00-7. (post-flight, in German)

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Co-Investigator(s): Megill, L. R. (Payload Manager and Contributor) (2), Busboso, E. (NASA Technical Manager) (3)
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Experiment Origin: Scotland
Mission: STS Launch #10, STS-011 (STS 41-B, Challenger)
Launch Date/Expt. Date: February 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) canister G-004
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of this Experiment Within G-004: University of Aberdeen, Scotland
Processing Facility: Capacitance micrometer with accompanying vibration isolation system
Builder of Processing Facility: University of Aberdeen Workshop, Aberdeen, Scotland

Experiment:
Stability of Materials - Residual Acceleration Measurements

This experiment was one of six investigations housed within the G-004 Get Away Special canister during STS-011. Three other experiments (of the six) were applicable to this data base (see Kitaura, STS-011 (Chapter 2); Thomas, S. (Chapter 12), STS-011; Gadsden (Brownian Motion), STS-011 (Chapter 15)). Although the objective of this experiment was not clearly presented, it appears that the investigation was designed to measure the very low-gravity (residual) accelerations experienced by the space shuttle.

<Note: Reference (3) gave a somewhat detailed summary of the experimental setup. Briefly, it stated that the setup consisted of (1) two flat strips of beryllium copper mounted in a frame, (2) detectors to record strip deflection, and (3) a vibration isolation system to isolate the assembly from high levels of shuttle vibration. The Principal Investigator indicated that this summary in Reference (3) was not correct, but did not then provide a detailed setup description. Instead, he simply stated that a capacitance micrometer was employed. No other information describing the setup could be located at this time.>

It was reported (and verified by the Principal Investigator) that "During the flight, the deflection detectors and associated electronics performed correctly, and deflections of the strips within the design range of the instrument were indeed recorded. Unfortunately, these appear to have the form of random vibration at the level of about 1/10,000 of a millimetre- which is around 1,000 times larger than the smallest deflections it was hoped to detect. At present it appears that, despite the success in gathering data, the quality has been much reduced by this excessive vibration-which possibly results from a failure of... [the] anti-vibration system to deploy correctly in orbit." (3, p. 3) <Note: As stated above, the nature of the deflectors and isolation system was not clearly provided by the Principal Investigator.>

Reference (3) indicated that further analysis of the data was to be performed and through this analysis the investigators hoped to recover "...some meaningful deflection measurements and to diagnose the failure in the vibration isolation technique." (3, p. 3)

No further information describing the results of this experiment could be located.

Key Words: Technological Experiments, Acceleration Measurements, Vibration Isolation Systems, Acceleration Effects

Number of Samples: two
Sample Materials: Unclear, possibly flat strips of beryllium-copper were used in the acceleration measurement device. <Note: When queried, the Principal Investigator did not indicate that this information was incorrect under the Sample Materials section, although he had indicated that related information was incorrect in the original experiment summary (see note above).>
Container Materials: not applicable

Experiment/Material Applications:
Although no applications of this research were cited in the applicable literature, residual (very low-gravity), very low frequency accelerations are suspected to be highly detrimental to several low-gravity fluids and materials processing initiatives. Measurement of these types of small accelerations is a difficult task and presents a challenge to investigators who wish to docu-

ment just how low these accelerations (and their associated frequencies) are.

References/Applicable Publications:

(1) Cargo Systems Manual: GAS Annex for STS-11, JSC-17645 Annex STS-11, December 2, 1983. (mention of experiment only; preflight)

(2) Student Gas Program Internal Document, Utah State University, Logan, Utah, 1984. (appears to be post flight)

(3) Letter from Michael Gadsden (Dept. of Natural Philosophy, Aberdeen University, Aberdeen, Scotland) to L. Rex Megill (Utah State University Student Gas Program Manager, Logan, Utah), which included a short report by M. A. Player entitled: Get Away Special; Stability of Materials Project-Summary of Results, July 11, 1984. (preliminary results of University of Aberdeen's G-004 experiments) (post-flight)

(4) Getaway Special (GAS) Payloads. In Goddard Space Flight Center's Engineering Newsletter, April 1984.

(5) STS-11 Getaway Special Payload Descriptions, NASA News, NASA GSFC, 1984.

(6) NASA Press Kit, Mission 41-B, p. 27.

(7) Ridenoure, R.: Gas Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(8) Input received from Experiment Investigator, July 1993.

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Co-Investigator(s): Unknown
Affiliation(s): (1,2) Consortium for Materials Development in Space (CMDS), University of Alabama, Huntsville, Alabama

Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Two Accelerometers: (1) a NASA Marshall Space Flight Center (MSFC) Miniature Electrostatic Accelerometer (MEA) and (2) a University of Alabama in Huntsville Accelerometer.
Builder of Processing Facility: (1) Although the MEA accelerometer was on loan from MSFC, it does not appear to have been built by MSFC. It was noted that the MEA employed 3 Kearfoot accelerometers. (2) This accelerometer was built by R. Newberry, University of Alabama, Huntsville, Alabama.

Experiment:
Accelerometer Measurements On Board Consort 1

The major objective of this Consort 1 experiment was to measure the gravity levels at two different rocket locations during the coasting phase of the rocket flight.

The rocket was configured with two accelerometer systems. The first accelerometer system, provided by NASA, Marshall Space Flight Center, was a derivative of a measuring system flown on STS-007 (the Low-G Accelerometer System (LGAS)). The system consisted of three accelerometers, placed in an orthogonal triad. The triad arrangement allowed measurements in three axes. The accelerometers were hinged pendulum, electrically restrained measuring devices maintained at a constant temperature throughout the flight to insure accelerometer stability. Theoretical resolution of the output was quoted as 1.1×10^{-7} g/bit change/s.

The second accelerometer system, provided by the Consortium for Materials Development in Space (CMDS), was newly developed for the Consort mission. The system consisted of "...a linear servo instrument with a nonwearing elastic suspension.... The accelerometer had an analog torque-balanced sensor with a fused quartz flexure, a permanent magnet torquer, a capacitive pickoff system, and self-contained servo electronics.... A temperature sensor is incorporated in the instrument." (4, p. 349) Theoretical resolution of the output was quoted as 1×10^{-6} g.

Reportedly, "Acceleration measurements from the Consort flight showed that operation of the experiments themselves generated appreciable accelerations. This was expected from mixing motors for experiments... that ran only during the first several seconds of free flight. However, motorized 35-mm cameras of standard commercial design generated acceleration spikes up to 0.1 g when exposures were made and the film was advanced. Acceleration measurements averaged over one second showed that the long-period acceleration environment attained the 10^{-5} g goal." (2, p. 3-4)

"...a constant, anomalous force was detected providing a constant acceleration throughout the low-g period of 7 X 10^{-5} g in the LGAS Y-axis and 8 X 10^{-5} g in the Z-axis. In the X-axis this force is below the measurement limit (<10^{-5} g). The composite residual force vector was about 10^{-4} g.... (2, p. 3)

"Since the roll motion [of the rocket] was very small and the CM [Center of Mass] was brought accurately to the center during spin balancing, there is no doubt that the measured accelerations are due to an anomalous force which could be residual off-gassing, an air leak through the non-sealed doors, leaking thrusters, or some other phenomenon.

"Finally, detection of the disturbances caused by operating the high temperature sintering furnace [see J. E. Smith, Consort 1 (Chapter 13)] were recorded by the LGAS package. The composite maximum force vector was close to the X-Z plane and was found to be consistent with design." (5, pp. 47-48) <Note: it is not clear what is meant by "consistent with design." No further information concerning this design was presented.>

Key Words: Technological Experiments, Acceleration Measurements, Accelerations/Vibrations Produced by Onboard Equipment, Rocket Motion, Acceleration Effects

Number of Samples: not applicable
Sample Materials: Accelerometer systems: "The first, the MEA Accelerometer Package or LGAS (Low-G Accelerometer System) on loan from NASA's Marshall Space Flight Center, uses three Kearfott 2412 accelerometers.... The output noise limit is 1 X 10^{-5} g and the bias u about 3 X 10^{-5} g.

"The second, a unit assembled by the CMDS... uses three QA-700 accelerometers.... Although the bias uncertainty of this system is high (8 X 10^{-3} g maximum) this system permits detection of rapidly oscillating disturbances...." (5, p. 47)

Container Materials: not applicable

Experiment/Material Applications:

"The two accelerometer systems are installed at different locations to be used not only for the purposes of best estimation of the gravity fields at different experiment locations but also for purposes of obtaining redundancy and gaining experience." (4, p. 349)

References/Applicable Publications:

(1) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronauitcal Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439, 11 pp. To be published in Acta Astronautica, 1990. (post-flight) <Note: The current publication status of this document is unclear.>

(2) Lundquist, C. A. and Wessling, F. C.: Microgravity Investigations on Suborbital Rockets. Presented at the IAF 40th International Astronautical Congress, October 7-13, Màlaga, Spain, IAF #89-425, 5 pp.

(3) Starfire 1/Consort 1 Post Flight Data and Final Summary Report, SSI-SF-600, April 28, 1989. (post-flight)

(4) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight)

(5) Measurement of the Microgravity Environment. In Consortium for Materials Development in Space, The University of Alabama in Huntsville, Annual Report, Technical Section, October 1, 1988-September 30, 1989, pp. 47-48. (post-flight)

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Co-Investigator(s): Boyd, W. C. (2)
Affiliation(s): (1,2) National Aeronautics and Space Administration (NASA), Johnson Space Flight Center (JSC), Houston, Texas

Experiment Origin: USA
Mission: STS Launch #13, STS-017 (STS 41-G, Challenger)
Launch Date/Expt. Date: October 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: On orbit propellant (hydrazine) resupply
Processing Facility: Fluid transfer system and controls for handling hazardous fluids on orbit
Builder of Processing Facility: Built internally at NASA Johnson Space Center, Houston, Texas

Experiment:
On Orbital Refueling System (ORS) Flight Demonstration

"Orbital refueling of satellites and other space vehicles... [may] be required for many future space programs. Many different technologies are involved in developing the capability to provide routine, safe, efficient on-orbit resupply of fluids: flow measurement, tank gauging, coupling design and standardization, etc. One key feature from a safety and operational standpoint relates to the compressive heating of the ullage gas in the tank as it is filled with liquid." (4, p. 3)

"The maximum temperature that the ullage gas can reach is that associated with adiabatic compression.... In practice there will be heat transfer from the gas to the vessel walls and surroundings, so the observed temperature rise will be less than that for true adiabatic expansion.

"A major potential hazard arises when certain fuels are transferred in this manner. Hydrazine, commonly used as a fuel on spacecraft, decomposes spontaneously around 200°F, with release of a great deal of energy. If a small amount of hydrazine accidentally leaked to the ullage side of the bladder, it could easily be heated above 200 °F by too-rapid filling of the tank. The ullage gas is heated by compression; it is cooled by heat transfer to the tank walls and, in turn, to the surroundings." (4, p. 4)

The Orbital Refueling System (ORS) was a low-cost flight experiment fabricated and certified in-house at NASA Johnson Space Center (JSC). The experiment laid the groundwork within NASA for the development of orbital fluid resupply tankers. It initiated and demonstrated the basic design concepts, operational guidelines, and operational procedures for handling hazardous

fluid transfers within the space shuttle payload bay by space-suited astronauts.

The specific objectives of the experiment included:

"(a) Demonstrate extravehicular activity (EVA) tool/valve interface for typical existing satellite propellant and pressurant servicing valves that were not designed for in-flight reservicing.

"(b) Demonstrate Orbiter-to-satellite interface for control of fluid transfer from the Orbiter aft flight deck. Establish procedures for transferring hydrazine in the payload bay.

"(c) Establish procedures for crew EVA operations on a hydrazine system with potential crew/Orbiter exposure to hydrazine.

"(d) Fabricate a system which, with minor modifications, could be reflown to permit orbital propellant refueling of a satellite such as LANDSAT. (4, p. 4-5)

The ORS was designed so that it would not pose any imminent hazard to the crew or mission in the event of any two simultaneous component failures. Since it was evident that the low-gravity heat transfer would have a major impact on the experiment, the maximum possible adiabatic temperature increase was calculated for each transfer. "Flow was stopped at the point where a purely adiabatic compression would have raised the ullage gas temperature 150 ^{o}F." (4, p. 5) (It was noted that obtaining heat transfer data was not a primary objective of the experiment.)

During the mission, liquid hydrazine fuel was transferred back and forth from one spherical bladder tank to another using pressurized nitrogen as the driving force. The "...experiment involved an extravehicular activity (EVA) during which the crew engaged a hydrazine connection between a simulated tanker and a simulation of a Landsat type propulsion system fluid interface. The Landsat uses 'standard' propellant servicing couplings designed for ground use. In order to safely connect the simulated tanker fluid line to these 'standard' couplings, a special set of tools... and procedures were developed at Johnson Space Center (JSC) to maintain a minimum of two seals between the crew and propellant at all times during the mating cycle. <Note: Although a detailed ORS fluid system schematic was provided in Reference (2) a more detailed (written) description of the ORS was not provided in the available References.> In addition to the propellant interface engagement, a total of 904 pounds of hydrazine was transferred in 6 transfers [285 minutes of flow]

between the simulated tanker and spacecraft to demonstrate control of ullage gas recompression temperatures during reservicing. <Note: The ullage is the amount that a container lacks of being full.>

"The fluid reservicing system was controlled by the STS crew from the orbiter aft flight deck using a dedicated ORS keyboard display unit (KDU) to communicate with the ORS computer.... This was the first use of this type of control system that could evolve into control of complex payloads without the overhead cost and schedule associated with control thru <sic> the orbiter general purpose computer (GPU)." (2, p. 2)

All transfers were carried out satisfactorily. Early analysis of the flight data indicated that the ullage compression process was much closer to an isothermal process than an adiabatic process. (Reference (4))

Key Words: Technological Experiments, Propellant Transfer, Refueling in Orbit, Liquid Transfer, Fluid Management, Contained Fluids, Partially Filled Containers, Free Surface, Solid/Liquid Interface, Liquid/Vapor Interface, Surface Tension, Heat Transfer, Compressive Heating, Adiabatic Expansion, Space Shuttle Safety

Number of Samples: one ORS system
Sample Materials: hydrazine
Container Materials: unknown

Experiment/Material Applications:
Hydrazine is often used as a spacecraft maneuvering propellant.

References/Applicable Publications:
(1) Input received from Experiment Investigator, July 1988.

(2) Griffin, J. W.: Background and Programmatic Approach for the Development of Orbital Fluid Resupply Tankers. AIAA/ASME/SAE/ASEE 22nd Joint Propulsion Conference, Huntsville, Alabama, June 16-18, 1986, AIAA Paper #86-1601, 7 pp. (post-flight)

(3) Input received from Principal Investigator J. W. Griffin, August 1993.

(4) Kauffman, D.: An Analysis of Ullage Heat Transfer in the Orbital Refueling System. Internal Note, Johnson Space Center JSC-20912, William C. Boyd, ed., October 21, 1985, 72 pp.

(5) Personal communication with Principal Investigator J. W. Griffin, September 1993.

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Co-Investigator(s): GM Vacuum Coating Laboratory (2)
Affiliation(s): (1) Marshall-McShane Designs, Prescott, Arizona; (2) Newport Beach, California

Experiment Origin: USA
Mission: STS Launch #13, STS-017 (STS 41-G, Challenger)
Launch Date/Expt. Date: October 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) Canister G-038
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-038: Joseph W. McShane, Prescott, Arizona/GM Vacuum Coating Laboratory, Newport Beach, California
Processing Facility: Glass sphere connected to space vacuum
Builder of Processing Facility: Unknown, possibly GM Vacuum Coating Laboratory, Newport Beach, California (The glass spheres used in the experiment were blown by the Schott Glass Works Company, Germany).

Experiment:
Art in Space: Sampling and Artistic Preservation of the Space Vacuum

"For the first time since man first gazed at the stars and from an earth-bound, one atmosphere, one-gravity perspective sought understanding of the unfathomable heavens through his art... [this experiment presented the opportunity] to make use of man's technology as an extension of the artist's eye and hand to venture forth directly into the vacuum and weightlessness of space, seeking understanding." (4, p. 120)

This experiment was one of three investigations housed within the G-038 Get Away Special (GAS) canister on STS-017. The two other experiments are described under McShane, STS-017 "Art in Space: Coating of Glass Spheres by Vacuum Deposition Techniques" (Chapter 10). The specific objective of the experiment reported here was to sample the space vacuum environment and artistically preserve that environment for return to Earth.

During the experiment, the interior of a 22000-ml glass sphere was exposed to the space vacuum environment via a high vacuum valve. "Over a three day period the interior of the sphere attained an equilibrium with the vacuum of the shuttle orbit, becoming one with the vacuum of space. A copper tube connecting the sphere to the valve was cold welded, permanently sealing the sphere, creating the sculpture "S.P.A.C.E.". Attached to the sphere is a Baratron capasitance [sic] manometer, [(]a vacuum gauge capable of a digital reading of the vacuum forming the

sculpture inside the sphere[)]." (5, p. 270)

It was noted by the investigator that "The sculpture "S.P.A.C.E." is not the glass, but the outer space contained within. The sphere serves only to keep the one-g atmosphere from intruding on the space within, creating an anomaly of our common experience; a sculpture to observe and stimulate wonder about the nature and meaning of space, a sculpture to touch and know that only an 1/8" of glass separates one from space." (5, p. 271)

<Note: Further details of the captured vacuum were not provided.>

Key Words: Technological Experiments, Art, Vacuum, Space Vacuum, Sampling of Space Vacuum, Direct Exposure to Space Environment, Welding

Number of Samples: one
Sample Materials: space vacuum
Container Materials: glass sphere

Experiment/Material Applications:
It appears that this arts-science payload was created to help man experience the mysteries of space.

References/Applicable Publications:

(1) Cargo Systems Manual: GAS Annex for STS-11, JSC-17645 Annex STS-11, December 2, 1983. (preflight; very short description)

(2) Space Shuttle Mission 41-G. NASA Press Kit, October 1984, pp. 23-24. (preflight; very short description)

(3) Cargo Systems Manual: GAS Annex for STS 41-G. JSC-17645 41-G, September 4, 1984. (short description; preflight)

(4) McShane, J. W. and Coursan, C. D.: An Artist's Exploration of Space. In NASA Goddard Space Flight Center's 1984 Get Away Special Symposium, NASA CP-2324, August 1-2, 1984, pp. 119-126. (preflight)

(5) McShane, J. W.: Art in Space -- A Divergent Exploration. In Goddard Space Flight Center's 1985 Get Away Special Experimenter's Symposium, October 8-9, 1985, pp. 267-273. NASA CP-2401. (post-flight)

(6) Shuttle Payload Creates Space Sculpture, AW&T, October 15, 1984.

(7) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, the NASA GAS Team, 1989, p. 23. (post-flight; very brief description)

(8) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report # EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special Canister mission history)

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Co-Investigator(s): None
Affiliation(s): (1,2) Vertical Horizons, Inc., Flushing, New York

Experiment Origin: USA
Mission: STS Launch #24, STS-032 (STS 61-C, Challenger)
Launch Date/Expt. Date: January 1986
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) Canister G-481
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay (carried in the standard NASA GAS Bridge Carrier System)
Primary Developer/Sponsor of G-481: Vertical Horizons, Flushing, New York
Processing Facility: Not specifically applicable. Raw, primed and painted samples of linen canvas, concentrically rolled in polyurethane foam were used as the experimental medium. Pressure in canister maintained at one atmosphere. A thermograph powered by 4 AA batteries recorded temperature changes inside canister at hourly intervals.
Builder of Processing Facility: Not applicable

Experiment:
Reaction of Oil Paints on Canvas to Space Travel

The specific objective of this STS-032 experiment was to determine the effects of vibration, temperature change, reduced gravity, and excessive g-stresses on fine arts materials.

Primed and unprimed linen samples, some of which were painted with oil colors using a wide variety of pigments, were employed as test materials. On Earth, the paints were applied using traditional artistic methods, creating actual paintings similar to those which may some day be transported or created on space flights.

The samples were rolled between layers of polyurethane foam and placed in the G-481 Get Away Special (GAS) canister. A thermograph was inserted into the center of the roll to record temperature changes within the can at hourly intervals.

After the 6-day mission, the samples were evaluated by several techniques including X-ray and ultraviolet examination. The following results were reported (2, p. 115):

"A. The linen and painted surfaces showed no sign of oxidation.*
B. The surfaces showed no accumulation of foreign substances.
C. The surface layers* were fully intact with no evidence of cracking* or flaking* of the pigments.

D. There was no sign of cupping or cleavage.*"

<Note: Terms followed by an asterisk (above) were further defined in the report.>

Reportedly, "Temperature changes within the G. A. S. canister were not recorded due to an error in the programming of the thermograph." (2, p. 115)

The investigators concluded that no degradation was apparent and that "...materials of the fine arts can be transported for limited periods of time into space and returned safely." (2, p. 115)

Key Words: Technological Experiments, Art, Paint on Canvas, Reaction of Oil Paints in Space, Coated Surfaces, Acceleration Effects, Oxidation, Surface Morphology, Surface Roughness, Hardware Malfunction

Number of Samples: 15
Sample Materials: Unprimed Belgium linen (three samples), single-primed Belgian linen (three samples), double-primed linen (three samples), single-primed Belgian linen painted with oil colors (three samples), double-primed Belgian linen painted with oil colors (three samples). The atmosphere of the can was similar to that of a typical spacecraft environment.
Container Materials: polyurethane foam in GAS canister

Experiment/Material Applications:
In anticipation of transportation and creation of fine art in space, this experiment allowed examination of artist materials packaged and exposed to the rigors of space flight.

References/Applicable Publications:
(1) Space Shuttle Mission 61-C, NASA Press Kit, December 1985, p. 17. (preflight)

(2) Kurtz, E. and Wishnow, H.: The Transportation of Fine Arts Materials Aboard the Space Shuttle Columbia. In the 1988 Get Away Special Experimenter's Symposium, Cocoa Beach Florida, September 27-29, 1988, NASA CP-3008, pp. 113-119. (post-flight)

(3) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Getaway Special Canister mission history)

(4) Input received from Principal Investigator, H. Wishnow, August 1989.

(5) Input received from Principal Investigator, E. Kurtz, June 1993.

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Principal Investigator(s): Casarez, C. (1)
Co-Investigator(s): Izquierdo, M. (Project Engineer) (2)
Affiliation(s): (1) During STS 51-G: Hanks High School, El Paso, Texas, Currently: Unknown; (2) El Paso Natural Gas, El Paso, Texas

Experiment Origin: USA
Mission: STS Launch #18, STS-025 (STS 51-G, Discovery)
Launch Date/Expt. Date: June 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: High School Student Experiment
NASA Get Away Special (GAS) Canister G-034
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-034: Texas High Schools (Ysleta and El Paso Districts)/Dickshire Coors, El Paso, Texas
Processing Facility: Liquid Dye Laser
Builder of Processing Facility: Unknown

Experiment:
Testing of a Liquid Laser

During operation of a liquid laser either in space or on Earth, (1) fluid "...is pushed through the optical cavity by means of a mechanical pump system..." (1, p. 68) and then (2) "[t]uning of the coherent beam is accomplished by the use of a prism or... [diffraction] grating... isolating only the part of the spectrum to be studied...." (1, p. 68)

This investigation was one of thirteen experiments housed within the G-034 Get Away Special Canister during the STS-025 mission. (Three other of the thirteen investigations are applicable to this data base (see Foster, STS-025 (Chapter 2); M. Moore, STS-025 (Chapter 8); Thurston, STS-025 (Chapter 18)).) The objective of this experiment was to compare liquid laser operation in the space environment with liquid laser operation on Earth.

Experimental system characteristics to be compared included a) the temperature of the liquid laser and the pump area, "...b) the condition of the beam in respect to color, intensity and distortion; [and] c) the relative power output of the laser...." (1, p. 68) The expected experimental procedure (which was only very briefly described in two sentences in Reference (1)) was unclear: "Bring the temperature up to 0° C and maintain at 0° C. Then follow procedures on flow chart for liquid laser project." (1, p. 63) <Note: An illustration of this flow chart was not provided.>

During the mission, a Plexiglas™ case, which enclosed another of the investigations housed within the Get Away Special canister (a seed germination experiment) broke "...spilling a water/formaldehyde mixture inside the GAS can. Several seconds later the batteries and/or controller shorted, ending all experiments except... [a wicking of fluids experiment (See Foster, STS 025)] which had its own power supply and controller." (4, p. 34)

Key Words: Technological Experiments, Liquid Lasers, Liquid Transfer, Liquid Leakage, Contamination Source, Battery Short

Number of Samples: Unknown; it appears that a single liquid laser was to be evaluated.
Sample Materials: liquid dye laser (liquid and dye unspecified)
Container Materials: unknown

Experiment/Material Applications:
Reportedly, because the "...laser has many physical uses in space..." (1, p. 63) characteristics of a liquid laser were to be evaluated.

References/Applicable Publications:

(1) El Paso & Ysleta Schools Get Away Special Payload #34. In Goddard Space Flight Center's 1984 Get Away Special Experimenter's Symposium, NASA CP-2324, August 1-2, 1984, pp. 59-68. (preflight)

(2) Cargo Systems Manual: Gas Annex for STS 51-G, JSC 17645 51-G, Rev. A, March 20, 1985. (very short description; preflight)

(3) NASA Space Shuttle Mission 51-G Press Kit. June 1985, p. 20. (very short description; preflight)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Company, Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Getaway Special Canister mission history)

(5) G-034 Payload Accommodations Requirements, NASA Goddard Space Flight Center, 1985.

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Principal Investigator(s): Thurston, J. (1)
Co-Investigator(s): Izquierdo, M. (Project Engineer) (2)
Affiliation(s): (1) During STS-025: Canyon Hills High School, Texas, Currently: Unknown; (2) El Paso Natural Gas, El Paso, Texas

Experiment Origin: USA
Mission: STS Launch #18, STS-025 (STS 51-G, Discovery)
Launch Date/Expt. Date: Not Applicable. (This experiment could not be readied in time for integration in to the GAS canister prior to the flight.)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: High School Student Experiment
NASA Get Away Special (GAS) canister G-034
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-034: Texas High Schools (Ysleta and El Paso Districts)/Dickshire Coors, El Paso, Texas
Processing Facility: Dynamic Random Access Memory (DRAM) Chips which consisted of thousands of transistors
Builder of Processing Facility: Japanese and American chips were to be employed. The specific brands were unspecified.

Experiment:
Performance of Dynamic Random Access Memory (DRAM) Chip in Space Environment

"...[Dynamic Random Access Memory] DRAMS consist of thousands of transistors in which the gates can be charged to a certain voltage level. Because the charge on the gates leaks away slowly, the charges have to be read and restored to their proper level periodically. This process is known as refresh. If refresh is not done within a certain time limit, the charge in the gates will have leaked away and any data stored in the chips will be lost." (1, p. 67)

This investigation was to be one of thirteen experiments housed within the G-034 Get Away Special canister on STS-025. (Three other of these thirteen investigations are applicable to this data base (see Casarez, STS-025 (Chpater 18); Foster, STS-025 (Chapter 2); M. Moore, STS-025 (Chapter 8)).) The specific objective of this experiment was to determine if "...conditions in space such as cosmic rays and weightlessness affect the performance of computer chips." (1, p. 66)

In order to achieve this objective, space performance of Dynamic Random Access Memory (DRAM) chips was to be compared with Earth DRAM chip performance. Reportedly, differences in the performance of Japanese-manufactured and American-manufactured chips

were to be evaluated from both the Earth and space experiments. The expected inflight procedure was described as follows: "Testing of the DRAMs will be done by using a microprocessor to write a test pattern to the chips and then count any errors that occur. A 2K EEPROM will be used to record the number of errors that occur. This testing will be done with different amounts of time between refresh cycles to determine how fast the charge is leaking away from the gates." (1, p. 67)

Reference (4) reported that this experiment was not ready for launch at the planned integration time and was thus was not included in the canister.

No further information concerning this experiment could be located.

Key Words: Technological Experiments, Computer Chip Performance, Cosmic Rays, Transistors, Computer Data Storage, Electric Field

Number of Samples: unspecified
Sample Materials: Japanese and American Dynamic Random Access Memory (DRAM) Chips
Container Materials: not applicable

Experiment/Material Applications:
Investigations of this type should contribute information to the expected performance of computer chips/computer systems in the space environment.

References/Applicable Publications:

(1) El Paso & Ysleta Schools Get Away Special Payload #34. In Goddard Space Flight Center's 1984 Get Away Special Experimenter's Symposium, NASA CP-2324, August 1-2, 1984, pp. 59-68. (preflight)

(2) Cargo Systems Manual: GAS Annex for STS 51-G, JSC 17645 51-G, Rev. A, March 20, 1985. (preflight; very short description)

(3) NASA Space Shuttle Mission 51-G Press Kit, June 1985, p. 20. (very short description; preflight)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(5) G-034 Payload Accommodations Requirements, NASA Goddard Space Flight Center, 1985.

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Principal Investigator(s): Alltech Associates, Inc. (1)
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Affiliation(s): (1) Deerfield, Illinois; (2) Alltech Associates, Deerfield, Illinois; (3) National Aeronautics and Space Administration (NASA), Goddard Space Flight Center (GSFC), Greenbelt, Maryland

Experiment Origin: USA
Mission: STS Launch #24, STS-032 (STS 61-C, Columbia)
Launch Date/Expt. Date: January 1986
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) Canister G-446
Volume of Canister: 2.5 cubic feet
Location of Canister: STS Payload Bay, (Standard NASA Bridge Carrier System)
Primary Developer/Sponsor of G-446: Alltech Associates, Inc., Deerfield, Illinois
Processing Facility: High Performance Liquid Chromatography (HPLC) analytical column manufacturing system
Builder of Processing Facility: Designed by Alltech Associates of Waukegan, Illinois

Experiment:
High Performance Liquid Chromatography (HPLC)

Few details of this STS Getaway Special Canister experiment could be located at this time. The STS 61-C press kit, which was released prior to the launch of the STS 61-C mission, briefly described the experiment objective and equipment setup. "The purpose of this experiment is to learn what effect gravity has on [the] particle dispersion of packing material in High Performance Liquid Chromatography (HPLC) analytical columns. Contained in a 2.5-ft., 60-lb. canister, the payload consists of an automated HPLC analytical column manufacturing system that will produce HPLC columns in microgravity. Post landing, the samples will be returned to Alltech Associates, Inc. for analysis." (1, p. 17)

Very little post-flight information could be located which described experimental results. Reference (2) reported that "...this experiment manufactured High Performance Liquid Chromatography Analytical Columns in microgravity. Used for chemical analysis, the columns allow the separation of a chemical mixture into its components, so the chemicals can be quantified. When manufactured on earth, the columns are not as efficient as theoretically possible, because minute particles with which they are packed do not settle uniformly. The experiment's designers expected that by reducing gravity, a more efficient column could be produced." (2, p. 35)

No further information concerning this experiment could be located.

Key Words: Technological Experiments, High Performance Liquid Chromatography, Particle Dispersion, Packing Material, Particle Separation, Separation of Components

Number of Samples: unknown
Sample Materials: unknown
Container Materials: unknown

Experiment/Material Applications:
Although it was stated that "Fields as varied as medicine, law enforcement, and petroleum processing could benefit from the results of G446," (2, p. 35) such benefits were not further detailed. It was expected that low-gravity production of the columns would result in a more uniform separation of the mixtures of interest.

References/Applicable Publications:

(1) NASA Space Shuttle Mission 61-C Press Kit, December 1985, p. 17. (very brief preflight summary)

(2) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division. The NASA GAS Team, 1989, p. 35. (does not discuss post-flight results)

(3) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11 October 2, 1987. (Get Away Special Canister mission history)

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Principal Investigator(s): Hebert, D. J. (1)
Co-Investigator(s): Lafferty, T. (Engineer and Science Representative) (2)
Affiliation(s): (1) Pre STS-032: Appleton High School West, Appleton, Wisconsin, During STS-032: US Naval Academy, Annapolis, MD, Currently: Physician, Regimental Aid, Kaneohe Bay, Hawaii; (2) James River Paper Corporation, Neenah, Wisconsin

Experiment Origin: USA
Mission: STS Launch #24, STS-032 (STS 61-C, Columbia)
Launch Date/Expt. Date: January 1986
Launched From: NASA Kennedy Space Center, Florida
Payload Type: High School Student Experiment, Shuttle Student Involvement Program (SSIP), STS Middeck Experiment
Processing Facility: Nine acrylic cylinders in which a fiber/slurry mixture was forced through a screen by a hand operated piston
Builder of Processing Facility: Designed by James River Paper Corporation, Neenah, Wisconsin; Built by Metal Products, Appleton, Wisconsin

Experiment:
A Study of Paper Fiber Formation (SE83-4)

During the formation of paper on Earth, the distribution of individual fibers in the slurry/fiber mixture and paper product is significantly affected by gravity-dependent forces. Thus, knowledge about the actual gravitational effects on fibers is valuable. Space-produced paper should exhibit more uniform fiber distribution than Earth-produced paper.

The specific objective of this STS high school student experiment was to study the reduced gravity formation of cellulose fibers in a fiber mat. This objective was to be achieved by examining paper forming fibers (1) as they are suspended in solution prior to distribution on a paper forming screen, and (2) after they are distributed on the paper forming screen.

Nine paper making apparatuses were prepared for the mission. All nine experiments were the same except for one variable. This variable was the addition of polymers to the fiber/slurry solution which promote or hinder fiber arrangements: the addition of anionic polymer increases floccing; the addition of cationic polymer prevents floccing.

Each apparatus was equipped with a 4-inch diameter cylindrical tank made of clear acrylic Plexiglas™ (total slurry volume .750 liter). Each of the acrylic tanks was fitted with a 100 mesh screen at one end of the cylinder, and equipped with a hand-

operated piston at the opposite end of the cylinder.

Prior to the mission, (1) tanks 1,2, and 3 were filled with a "control" fiber/slurry mixture (no chemical additives) containing .125% short fibers, .125% long fibers, and 99.9975% water, (2) tanks 4, 5, and 6 were filled with the control fiber/slurry mixture plus cationic polymer (500 parts cationic polymer per million parts of water), and (3) tanks 7, 8, and 9 were filled with the control fiber/slurry mixture plus anionic polymer (500 parts anionic polymer per million parts water). <Note: See the materials section below for additional information.>

During the mission, Mission Specialist Steve Hawley "...agitated the fiber slurry by rotating the cylinder apparatus and then while holding the unit static for 30 seconds, observed and photographed the fibers as they drifted in the aqueous solution." (2, p. 32) He then depressed the piston forcing the fiber/slurry through the cylinder and distributed the paper fibers on the screen at the opposite end. The liquid pushed through the screen exited the cylinder through an external tube behind the screen and was routed to the opposite end of the tank where it re-entered the cylinder chamber behind the piston. The fibers deposited into the screen were retained by the piston. The cylinders were then sealed until they could be opened at the James River Paper Company. (Each paper-forming apparatus was a closed sealable system, safe for operation in the shuttle.) Once the cylinders were returned to James River, the the screens were removed from the cylinders and dried.

While the experiments were being performed in space, nine identical units at James River were employed to produce nine "Earth control" sheets. The sheets were then tested and compared with the space paper.

Non-destructive physical testing at James River (Neenah Technical Center) and the Institute of Paper Chemistry (Appleton, Wisconsin) was performed in order to preserve the samples. "The basis weight, thickness and Sheffield smoothness were measured using standard TAPPI procedures with the apparent density calculated from basis weight and thickness. Strength was evaluated by means of Sonic Modulus Testing at the Institute of Paper Chemistry. This device measures sound-wave velocities traveling through the sample. The square of the measured velocity, km^2/sec^2, can then be correlated with sheet strength properties....

"It was necessary to select a formation measurement device which could be used on small sheets (4-inch diameter) to indicate differences between macro-and micro-formation. The M/K Systems On-Line Formation Tester was selected. This instrument calculates

an average of 10,000 micro-opacity measurements in a 15-second test period. The 'formation number' is the voltage proportional to the mean peak-to-peak variation in light transmission during the 15-second test period....

"These formation test results show a statistically significant difference between the space made and the Earth-made sheets. All space-made sheets had lower (better) formation values than their Earth-made counterparts. This difference increased when the testing speed increased suggesting a better micro-formation for the space-made paper. This difference can be seen visually.

"Equal values in the absolute strength of space-made and Earth-made papers support the concept of relatively equal macro-formation. A lower strength standard deviation in the space-made paper supports the observation of improved micro-formation." (3, p. 7)

<Note: No discussion concerning the effect of the cationic and anionic polymers was presented. However, a table presented in Reference (3) appears to indicate that (in general) both in space and on Earth, the paper formed from a slurry with no additives exhibited the lowest (best) paper formation values (see Reference (3), Table II, page 7).>

It was concluded that the space-produced, hand-made sheets can have superior micro-formation compared to Earth-formed papers. It was reported, however, that it is not so clear "...as to why differences in formation exist or which factors may be contributing to the observed effects. Formation studies as reported in the literature generally have involved machine made papers and have not been concerned with the presence or absence of gravitational forces during the sheet forming process...."

"A concise explanation was offered by Van den Akker... who observed that 'not all fibers are of the same apparent density. In addition to natural variation within one species, there are differences between species and different densities result also from air entrapment within the lumen. In the Earth-made paper, the lighter fibers have a tendency to rise and the heavier ones to sink. This relative movement can cause minor fiber entanglement and some loss in micro-formation. This phenomena would be reduced in space since gravitational forces would not effect relative movement of fibers of different density'. This is supported by an earlier experiment in which the absence of hydrostatic pressure in zero-gravity accounted for reduced buoyancy forces, resulting in the elimination of natural convection and sedimentation effects.... <Note: This "earlier experiment" was not identified.> Therefore, it appears that eliminating the relative movement between the fibers has effectively reduced the

flocculating rate or tendency in the fiber system such that improved formation could be both instrumentally measured and visually observed." (3, pp. 7-8)

It was recommended that "Researchers who want to produce more uniform handsheets need to minimize differences in fiber density and fiber sink rates or form the sheet in the shortest practical time after agitation stops or continue agitation during the formation process." (3, p. 8)

<Note: No post-flight discussion of the fiber distribution within the slurry prior to piston depression could be located.>

Key Words: Technological Experiments, Paper Formation, Fibers, Fiber Dispersions, Particle Dispersion, Particle Distribution, Suspension of Particles, Homogeneous Dispersion, Sedimentation, Aqueous Solutions, Slurry Solutions, Polymers, Piston System, Liquid Transfer, Liquid Mixing, Sample Rotation, Material Strength, Hydrostatic Pressure, Buoyancy Forces, Density Difference, Contained Fluids, Liquid Reservoir

Number of Samples: nine
Sample Materials: Reference (2) reported the following: (1) tanks 1, 2, and 3 were filled with a "control" fiber/slurry mixture (no chemical additives) containing 0.125% short fibers, 0.125% long fibers, and 99.9975% water; (2) tanks 4, 5, and 6 were filled with the control fiber/slurry mixture plus cationic polymer (500 parts cationic polymer per million parts of water); and (3) tanks 7, 8, and 9 were filled with the control fiber/slurry mixture plus anionic polymer (500 parts anionic polymer per million parts water). Reference (3) reported the following: Three of the paper-forming apparatuses contained 1/1 blend of bleached, unrefined hardwood kraft pulp and bleached, unrefined softwood kraft pulp uniformly mixed and diluted to 0.07% consistency. Three of paper forming apparatuses employed this diluted blend plus 50 ppm of cationic polymer, and three of the paper-forming apparatuses contained the diluted blend plus 50 ppm of anionic polymer.
Container Materials: Plexiglas™ acrylic

Experiment/Material Applications:
During the formation of paper on Earth, heavier fibers separate from lighter fibers, thus affecting the distribution of the fibers in the final paper. If these fibers could be evenly distributed, the paper may be of extremely high printing quality. Space-produced paper should exhibit more uniform fiber distribution than Earth-produced paper.

References/Applicable Publications:

(1) Space Shuttle Mission 61-C. NASA Press Kit, December 1985, p. 20. (preflight)

(2) Lafferty, T.: Papermaking in Space: Technology Can't Get Any Higher Than This! PIMA (The Magazine for Papermaking Professionals), February 1986, pp. 30-33. (post-flight)

(3) Hebert, D., Lafferty, T., and Thorp, B.: Space-Formed Paper Properties. Document received from Co-Investigator T. Lafferty, December 1993, 10 pp. (post-flight)

(4) Input received from Co-Investigator, T. Lafferty, 1993.

(5) Study of Paper Formation in Microgravity. In Shuttle Student Involvement Program (SSIP) Final Reports of Experiments Flown, NASA/JSC Internal Note, JSC 24005, October 20, 1989.

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REPORT DOCUMENTATION PAGE	Form Approved OMB No. 0704-0188

1. AGENCY USE ONLY (Leave Blank)	2. REPORT DATE November 1996	3. REPORT TYPE AND DATES COVERED Technical Memorandum

4. TITLE AND SUBTITLE

The Microgravity Research Experiments (MICREX) Data Base

5. FUNDING NUMBERS

6. AUTHOR(S)

C.A. Winter and J.C. Jones*

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama 35812

8. PERFORMING ORGANIZATON REPORT NUMBERS
TM-108523
Volume IV

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

National Aeronautics and Space Administration
Washington, DC 20546-0001

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

Prepared by Space Sciences Laboratory, Science and Engineering Directorate
*University of Alabama in Huntsville (UAH)

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Unclassified - Unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

An electronic data base identifying over 800 fluids and materials processing experiments performed in a low-gravity environment has been created at NASA Marshall Space Flight Center. The compilation, called MICREX (MICrogravity Research Experiments), was designed to document all such experimental efforts performed (1) on U.S. manned space vehicles, (2) on payloads deployed from U.S. manned space vehicles, and (3) on all domestic and international sounding rockets (excluding those of China and the former U.S.S.R.). Data available on most experiments include (1) principal and co-investigators (2) low-gravity mission, (3) processing facility, (4) experimental objectives and results, (5) identifying key words, (6) sample materials, (7) applications of the processed materials/research area, (8) experiment descriptive publications, and (9) contacts for more information concerning the experiment. This technical memorandum (1) summarizes the historical interest in reduced-gravity fluid dynamics, (2) describes the importance of a low-gravity fluids and materials processing data base, (4) describes the MICREX data base format and computational World Wide Web access procedures, and (5) documents (in hard-copy form) the descriptions of the first 600 fluids and materials processing experiments entered into MICREX.

14. SUBJECT TERMS data base, low-gravity materials processing, fluid dynamic experiments, low-g facilities, early rocket design, aircraft low-g test-beds, drop tubes, drop towers, sounding rockets, Apollo program, Skylab, space shuttle, low-g experiment history	15. NUMBER OF PAGES 507 16. PRICE CODE NTIS

17. SECURITY CLASSIFICATION	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	Unlimited

APPROVAL

The Microgravity Research Experiments (MICREX) Data Base

By C. A. Winter and J. C. Jones

The Information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

Gregory S. Wilson
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Space Sciences Laboratory

☆ U.S. GOVERNMENT PRINTING OFFICE 19 97–532-128/0003